IN VITRO WEAR OF GLASS-IONOMER CONTAINING RESTORATIVE MATERIALS

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ABSTRACT

Arthur Valeri: In vitro wear of glass-ionomer
containing restorative materials
(Under the direction of Terry Donovan)

This study compared in vitro wear of contemporary glass-ionomer containing dental materials commercially advertised for use in the permanent dentition as load-bearing restorations in a chewing simulator. Resin composite was tested as a control.

Four restorative dental materials were used in this study. Ionolux (VOCO America Inc.) is a resin-modified glass ionomer. Activa Bioactive Restorative (Pulpdent) is a bioactive ionic resin with reactive glass filler. Equia Forte HT and Equia Coat (GC America Inc.) is a high viscosity glass-ionomer hybrid system. Filtek Supreme Ultra (3M ESPE) is a visible light-activated resin composite.

After an estimated two years of clinical service, there was a statistically significant difference in mean volumetric wear for Activa Bioactive Restorative ($P=0.0081$, 95% CI: 0.3973, 0.4982) and Equia Forte HT ($P<0.001$, 95% CI: 1.2495, 1.8493), but no statistically significant difference in mean volumetric wear for Ionolux ($P=0.6653$) compared to control. Activa Bioactive Restorative wore approximately 60% less than, and Equia Forte HT twice more than Filtek Supreme Ultra on average, respectively. Clinical advantages of Activa Bioactive Restorative remain unknown. The resin-modified glass-ionomer Ionolux should be evaluated for further merit. The glass-ionomer hybrid system Equia Forte HT will likely experience unacceptable in vivo wear.

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Hz	Hertz
MPa	Megapascal
N	Newton(s)
SEM	Scanning electron microscopy

CHAPTER 1: REVIEW OF THE LITERATURE

1. Introduction and background

Variations of contemporary glass-ionomer containing dental materials are currently advertised for use as load-bearing permanent or semi-permanent direct restorations. Relatively limited information is available regarding laboratory or clinical performance of these new materials in comparison to resin composites or dental amalgam. Historically, glass-ionomer containing restorative materials are contraindicated in stress-bearing areas due to inferior mechanical properties, including high wear rates. Generations of glass-ionomer containing restorative materials, past and present, have provided the dental industry with a wide and potentially expanding scope of clinical indications. Modern unique and proprietary formulations of glass-ionomer containing restorative materials contain matrix or filler modifications compared to conventional formulations. Technology, including chewing simulators, enable partial imitation of the oral environment. Simulations that evaluate wear as a result of in vitro fatigue via cyclic loading and thermal cycling provides an economical method to identify those restorative dental materials with merit in preparation for resource-consuming clinical trials or product release.

2. Literature review

A review of the literature will identify the history and clinical performance of both conventional and contemporary generations of glass-ionomer containing restorative materials. Key chemical compositions, physical and mechanical properties will be reviewed in addition to concepts of restorative material retention via mechanical design and adhesive bonding. Finally, a

review of wear characteristics, mechanisms and in vitro fatigue test simulations and correlation to clinical performance will be described.

2.1. History of the glass-ionomer

Conventional glass-ionomer cement was invented by Wilson and Kent in 1969 as a new class of dental material and properly termed aluminosilicate polyacrylate. A chemical reaction following the combination of powdered fluoroaluminosilicate glass and aqueous solutions of polyacrylic acids hardened to form a composite with glass particulate filler reinforcing an aluminum phosphate gel[1].

2.1.1 Classifications and nomenclature of glass-ionomer cement

Classification of the glass-ionomer cement by Wilson and McLean was originally described by the manner in which the material was applied, most notably characterized by alteration of the liquid-powder ratio[2]. Although various modifications to this classification system arose and became more complex over time, the original descriptions retain contemporary relevance.

Table 1. Original classification of glass-ionomer cement according to Wilson and McLean.

Classification of Glass-Ionomer	Description
Type I	Luting cement
Type II	Restorative
Type III	Lining cement

Further generations of glass-ionomer containing restorative materials complicated nomenclature efforts based on hybrid formulations, such as those with photo- or chemical cure

polymerization reactions within the matrix as mixed with components of resin composites. As a matter of clarification, the term “glass-ionomer” is indicated when an acid-decomposable glass and a water-soluble acid set by a neutralization reaction, or alternatively “glass polyalkenoate cement” according to the International Organization for Standardization[3]. The term “resin-modified glass-ionomer” is indicated with the addition of photo- or chemical resin polymerization potential supplementary to the acid-base reaction of the glass-ionomer. In the absence of an acid-base reaction but presence of pre-reacted glass-ionomer as filler particles within a matrix, the term “polyacid-modified composite resin” or “compomer” is indicated. Other variations, including those with amalgam particles, ceramic fillers and more variables led to a diverse palate of terminology for glass-ionomer containing restorative materials[4]. As a result, a true spectrum of dental materials with pure glass-ionomers at one end and resin composites at the other is available to restorative dentistry[5].

2.1.2. Clinical indications and performance

Glass-ionomer containing restorative materials have a variety of potential clinical indications: lining or base restorations, a substructure for other direct or indirect dental restorations via the “sandwich” technique, provisional, semi-permanent or permanent restorations in the primary and permanent dentition, root-end restorations or perforation repair in endodontic surgery and pit and fissure sealants. Additional clinical applications include use as a luting agent for indirect coronal restorations and cementation of orthodontic bands.

2.1.2.1. Direct restorations

Direct restorations are those placed into a cavity to return the damaged or deformed tooth to form, function or facilitate cleansability of tooth surfaces. Additional consideration for esthetics towards patient satisfaction is often a requirement. Conventional nomenclature to

describe direct restoration type is based on cavity location and preparation design, from class I, II, III, etc. restorations[6]. Glass-ionomer containing restorations can be judiciously utilized as an interim or definitive restorative dental material for the moderate or high-caries risk patients as part of a disease control phase of treatment planning or other certain clinical situations[7].

2.1.2.1.1. Lining or base restorations

Cavity liners and bases, while both adjunct procedures of restorative dentistry, may be composed of the same material but are classified according to respective clinical application. Materials placed for bulk replacement of lost dentin or to block out undercut areas are considered base restorations. Cavity liners are typically a cement or resin coating less than 0.5 millimeters in depth and serve as a physical barrier to bacteria and their products while possibly providing a therapeutic effect[8]. Adequate dentinal thickness is important for maintaining tooth vitality and protecting the vital pulp[9].

Glass-ionomer containing restorative materials, in multiple varieties, may be clinically indicated for both lining and base restorations. Generally, toxic effects on dental pulp is the result of bacteria and bacterial byproducts in contrast to most restorative materials[10]. Conventional and resin-modified glass-ionomer cements provide an excellent bacterial seal and display good biocompatibility when used in close approximation but not direct contact with pulpal tissues[11].

2.1.2.1.2. Class V lesions

Clinical trials have consistently demonstrated glass-ionomer containing restorative materials survive at higher rates in comparison to resin composites in class V restorations[12,13]. However, decreased esthetic potential of glass-ionomers compared to resin composites remains a limitation[14].

Perhaps owing to decreased technique sensitivity, glass-ionomer containing restorative materials were observed to achieve the lowest annual failure rates when comparing all methods of adhesive protocols, including three-step etch-and-rinse, two-step etch-and-rinse, two-step self-etch, one-step self-etch techniques and polyacrylic acid conditioning[15]. While attempts to facilitate ease of adhesive bonding protocol for resin composites tend to decrease clinical effectiveness[16], the clinical steps required for glass-ionomer containing restorative materials in class V lesions are relatively fewer than resin composites alternatives, which require dentin bonding protocols that vary significantly. Other potential explanations for glass-ionomer restoration survival rates as cervical restorations are chemical bond to tooth structure, coefficient of thermal expansion and modulus of elasticity similarities between the material and tooth[17].

2.1.2.1.3. Sandwich technique

Restorative margins terminating on sub-gingival or dentinal substrates present multiple challenges, including field isolation, to traditional dental adhesive bonding protocols. In an attempt to increase predictability and utilize advantageous properties of glass-ionomers in dentinal bonding, the "sandwich" technique was proposed[18]. In theory, the glass-ionomer containing restorative material can be exposed at the deepest portion of the gingival margin to facilitate chemical bond and seal to tooth structure while simultaneously serving as a fluoride reservoir in direct contact with the oral environment. Equivocal evidence is available regarding clinical performance: Some suggest the seal may be worse with multiple restorative interfaces[19], whereas other evidence suggests the opposite[20] using microleakage as evidence to evaluate marginal seal. It is not proven whether or not evidence of microleakage is an indicator of clinical success, as the clinical relevance of microleakage studies are potentially misleading[21]. Overall, combining glass-ionomer cement and a resin composite in a single restoration may offer the

clinician advantages of each material[22]. Summarily, the sandwich technique is proposed with caution as there is not convincing, robust or high-quality evidence of clinical efficacy at this time.

2.1.2.1.4. Load-bearing restorations

Clinical trials involving glass-ionomers reveal poor performance as load-bearing restorations in the permanent dentition. When used as limited, single-surface occlusal restorations, glass ionomer containing restorations may perform satisfactory in the short-term; however, failure rates precipitously increase with the number of restored surfaces, especially interproximal areas[23,24,25,26].

One potential method of overcoming weaknesses of glass-ionomer restorative materials under forces of mastication is the concept of resin-glass hybrid systems, include the addition of a resin coating on the exposed surfaces of the glass-ionomer. When used in combination with a resin coating, performance of load-bearing glass-ionomer restoratives may or may not be enhanced. Clinical trials indicate performance up to two years may be acceptable in comparison to resin composites, but an unacceptable number of short-term failures of glass-ionomer hybrid systems persists[27,28]. However, limited evidence exists to demonstrate clinical performance up to 10 years demonstrating similar risks to increased number of restored surfaces[29]. Generally, high quality evidence is lacking at this time.

2.1.2.1.5. Atraumatic restorative technique

Atraumatic restorative technique involves caries excavation using solely hand instrumentation, typically without the use of local anesthetic and in combination with sealing pits and fissures. The caries removal is followed by placement of a glass-ionomer containing restorative material. This restorative technique is attuned towards field dental operations in areas

of limited resources or access to care[30]. Typically, restoration survival is prioritized over restoration success given the clinical limitations when atraumatic restorative technique is involved as a means to arrest caries activity in populations without adequate access to dental care. Restoration survival implies the restoration remains in situ, while restoration success would be defined by a scale such as United States Public Health Service or Ryge criteria[31]. Anatomic contour, color match and surface texture deficiencies either as a result of placement or following occlusal wear are typical compromises made in exchange for attempting to arrest or delay caries progression with minimal cost and resources as in the atraumatic restorative technique. Even with limited equipment, large restorations placed using the atraumatic restorative technique can demonstrate survival exceeding 95% after two years depending on the glass-ionomer material used[32]. Longer-term clinical studies indicate failure rates may increase to nearly 58% over a 10-year period, but insufficient information is available[33]. Regardless, the limited number of studies provide generally low-quality evidence with high risk of bias overall regarding this technique compared to conventional treatment, representing a dichotomous approach to restorative dentistry compared to clinical-based settings[34].

2.1.2.1.6. Pediatric dentistry

Conventional glass-ionomer cements demonstrate high failure rates in class II restorations in the primary dentition and are not recommended as load-bearing restorations[35]. However, resin-modified glass-ionomers can be successful depending on the size of the lesion[36]. Evaluation of load-bearing restorations in the primary dentition revealed resin-modified glass-ionomer multi-surface restorations survived comparable to resin composites up to a two-year period, but with pronounced occlusal wear a frequent observation[37]. Limited expected duration

of clinical performance due to natural exfoliation may provide a basis for resin-modified glass-ionomers as load-bearing restorations in the primary dentition.

2.1.2.1.7. Root-end restorations in endodontics

Root-end filling material selection is a considering during retrograde endodontic therapy. Many options in materials selection exist to create an artificial apical seal in a prepared root apex, including mineral trioxide aggregate, intermediate restorative material, super ethoxybenzoid acid, resin composite, glass-ionomers, and amalgam; limited available evidence is currently insufficient to conclude superiority of any material over another[38].

2.1.2.1.8. Pit and fissure sealants

Caries in permanent posterior teeth maintain the highest incidence and prevalence in anatomical pits and fissures[39]. A preventive treatment intervention to directly address this pathology includes pit and fissure sealants. The intent of the sealant is to prevent cariogenic bacteria, nutrients and subsequent byproducts from entering non-hygienic and caries-susceptible areas of the tooth[40]. Preventing these carious lesions through the use of glass-ionomer based sealants may be preferred over resin sealants when moisture contamination is a concern[41].

2.1.2.2. Luting agents

Glass-ionomer containing materials have long served as luting agents for indirect restorations and orthodontic appliance adhesion. Indirect restorations are fabricated extraorally and clinically delivered to the tooth or implant substrate surface. Orthodontic appliances, cemented to tooth surfaces, typically include brackets and bands attached to the unprepared tooth surface.

2.1.2.2.1. Indirect restorations

Glass-ionomers have an established track record for successful use as a dental cement. In order to facilitate enhanced indirect restoration seating, an oversized die is referenced for restoration fabrication leading to space between the tooth and the intaglio surface of the restoration[42]. The resultant space is filled by a luting agent. Glass-ionomer cements are able to achieve thicknesses under 25 micrometers. Glass-ionomer containing luting agents may not be the material of choice for implant restorations, as removing excess cement and peri-implant disease have been associated with their use[43]. Titanium surfaces may be damaged as part of the acid-base setting reaction or components associated with glass-ionomers[44].

2.1.2.2.2. Orthodontics

Application of orthodontic bands is with glass-ionomer containing cements is a potential option for clinicians, however there is insufficient evidence to suggest any clinically superior advantage of one particular material[45].

2.2. Chemistry and setting reaction

Conventional glass-ionomer cement is a combination of liquid and powder[46]. Glass-ionomers are composed of a cross-linked polyacid matrix with embedded glass particles. The mixture undergoes a neutralization reaction in water to form a salt that serves as a bound matrix. Generally, unreacted fluoroaluminosilicate glass serves as filler particles suspended in a polyacid copolymer matrix formed between high molecular weight acids with multiple functional groups.

There are generally three phases of the setting mechanism: Ion release, matrix formation, and polysalt maturation[47]. Upon mixing the liquid and powder, the hydrogen ions of polyalkenoic acid attack glass filler particles and release a cation, typically calcium or aluminum. Exposed carboxylic acid functional groups form ionic bond with these cations, subsequently

forming a hydrogel matrix[48]. The mixture is acidic upon placement, but within the first 24 hours the pH becomes more neutral[49]. The matrix is subject to continuous maturation over time.

2.2.1. Chemical composition

Conventional glass-ionomers contained a liquid and a powder. Modifications to original formulations include both concentrations of original components as well as additions to both the liquid and the powder.

2.2.1.1. Liquid

The liquid component consists predominantly of polyacrylic acids and water. Any number of various polycarboxylic acids including but not limited to tartaric, maleic and itaconic acids are useful in glass-ionomer cement formation[50].

2.2.1.2. Powder

The powder is industrially produced by forming a glass base consisting of high temperature 1100 to 5300 degrees Celsius fusing of quartz, alumina, cryolite, fluorite, aluminum trifluoride and aluminum phosphate with an overall a predominance of calcium or strontium aluminosilicates and fluoride. The homogenous melt is quenched to produce a glassy frit. Ground glass particles under 50 micrometers in length are then produced via milling, grinding and meshing[51].

2.2.2. Matrix, filler and chemical modifications

Many attempts to increase physical properties of glass-ionomer containing restorative materials began with filler modifications and changing the powder-liquid ratio. Changing the formulation of powder-liquid ratio at concentrations greater than 3.6 to one is characteristic of the so-called “high viscosity” glass-ionomer[52]. Other variations include the resin-modified glass-

ionomer and metal-modified glass-ionomers or attempts to incorporate components or pre-reacted glass-ionomers into the chemical composition of the restorative material.

2.2.2.1. Resin-modified glass-ionomer

With the addition of hydroxyethylmethacrylate and other components associated with resin composites with an activating agent such as camphorquinone the glass-ionomer is appropriately termed the resin-modified glass-ionomer. Introduced by Mitra, this formulation represented another major breakthrough in dental materials science. In general, resin-modified glass-ionomers have increased mechanical properties compared to conventional glass-ionomers[53].

2.2.2.2. Other variations

Contemporary variations of glass-ionomer containing restorations are not clearly described using conventional definitions, terminology, and classifications because of proprietary formulations leaving room for debate of the actual material composition and chemistry. New terms, largely provided by manufacturers, make comparison between products for the dental practitioner complicated. Some of the latest availabilities include: nanoparticle ceramics, so-called bioactive glass, fiber reinforced glass, amino acid additions and other methods to reinforce the material[54, 55]. Even more selections include so-called ionic resin composites without bisphenol A or bisphenol A glycidyl metaacrylate, which are advertised as “shock-absorbing” components with little scientific explanations in the event of Activa Bioactive Restorative[56]. Perhaps guarding trade secrets, manufacturers indeed retain legal rights to limit disclosure of proprietary formulations. Resultantly, recent meta-analysis reveals current nomenclature for the wide variety of direct restorative materials across the dental industry is insufficient and potentially affecting our ability to consolidate and interpret data[57].

2.2.3. Surface coatings

Early formulations of glass-ionomers were especially susceptible to degradation due to dehydration or excessive fluid contamination during matrix maturation resulting in compromised physical and mechanical properties. Efforts to prevent dehydration included the use of surface coatings, such as petroleum jelly or resin coatings.

When applied directly over glass-ionomer containing restorative materials placed as load-bearing restorations, some evidence suggests the type of surface coating has a direct effect on wear resistance. Unfilled or lightly filled resin coatings have less resistance to occlusal wear compared to higher-filled alternatives[58]. A resin coating also increases the flexural strength, fracture toughness and knoop hardness[59, 60].

2.2.4. Powder-liquid ratio

Altering the powder-liquid ratio of glass-ionomer cements affects the properties of the resultant polygel. Working time, setting time, and solubility are decreased while consistency, surface hardness and compressive strength are increased.

Table 2. Effect of powder-liquid ratio change on physical and mechanical properties.

Property	Effect of increased powder-liquid ratio
Setting time	Decreased
Solubility	Decreased
Working time	Decreased
Compressive strength	Increased
Consistency	Increased
Surface hardness	Increased

In general, the highest ratio possible while maintaining adequate working time is desired[61]. Altering the powder-liquid ratio is not clinically indicated for contemporary formulations given the availability of pre-measured delivery methods.

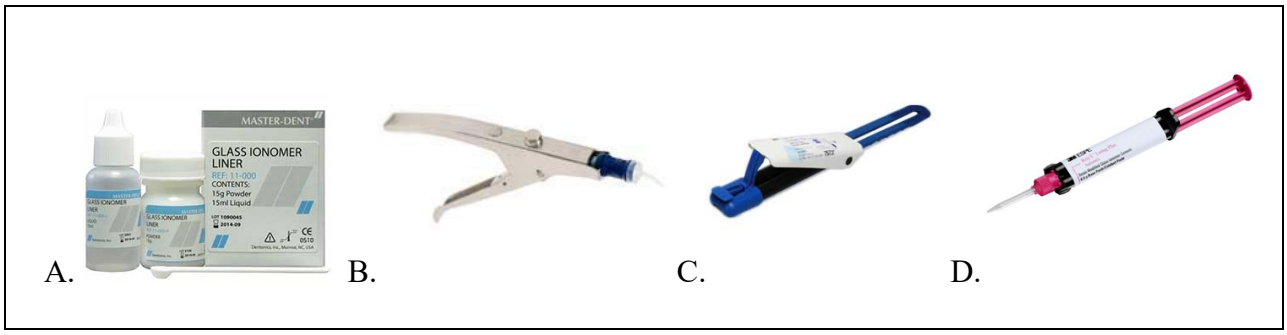
2.2.5. Mixing, setting and working time

Depending on the delivery method of glass-ionomer containing restorative materials, mixing, setting and working time varies. Working time is the amount of time available to the operator to manipulate and place the material. The presence of tartaric acid assists in decreasing the overall setting rate of conventional glass-ionomers while maintaining working time[62]. Resin-modified glass-ionomers will set upon photopolymerization, resin self-polymerization or acid-base glass-ionomer reaction, whichever occurs first depending on the product-specific chemistry. In general, longer working times can be advantageous to afford the operator opportunities to improve adaptation into prepared cavities, facilitate shaping of the material into proper form, remove excess material, etc. Photo-activated resin composites may have unlimited working time so long as excitatory wavelengths are avoided.

2.2.5.1. Delivery methods

Glass-ionomer containing restorative materials are packaged in two main categories: Powder-liquid and paste-paste. Powder-liquid is available in separate bottles or pre-measured, self-contained triturable capsules. Paste-paste systems include dispensable clicker and automix tubes which may or may not require a manufacturer-specific dispensing device.

Fig 1. A. Bottles. B. Capsules. C. Dispensable clicker. D. Automix tubes.



Contemporary evidence of dental cements across a complete spectrum of materials including glass-ionomers, resins and others, indicate that physical and mechanical properties vary as a direct result these modes of delivery, but the clinical significance of these differences as luting agents or cements remains unknown[63].

2.2.6. Matrix maturation

The glass-ionomer polygel matrix will continue to change after the initial setting reaction in a process called matrix maturation. This process can generally be separated into the short and long term phases[64].

2.2.6.1. Short versus long term maturation

The specific molecular weight, size and shape of organic polymers create potential for additional hydrogen bonds within the material. Pendant chains further increase molecular interaction potential. Although the majority of hardened polygel matrix formation of the glass-ionomers is essentially complete by 24 hours, changes in physical properties over a one-year period suggest the matrix undergoes continuous maturation, possibly the result of cation exchanges[65].

2.3. Restoration retention

Methods to prevent dislodgement of restorative materials are generally of two main strategies. The first is through mechanical means, which includes macro-retentive preparation design and micro-retentive surface conditioning via acid etching of tooth substrates. The second is through chemical, or hydrophilic, bonding to minerals tooth structure. The overarching goal of dental adhesion is achieving intimate adaptation of restorative material and tooth substrate[66].

Mechanical retention requires the use of undercut areas to prevent displacement of the material along paths of draw. This is achieved either macroscopically or microscopically. Macroscopic methods generally refer to preparation design of opposing converging walls with narrower dimensions in outer portions. Microscopic methods utilizes the concept of dental adhesion. Buonocore is credited with expanding the potential of adhesive dentistry through the acid-etched enamel technique, drastically increasing micromechanical potential on the tooth surface[67]. Using phosphoric acid, approximately ten micrometers of enamel are removed and another 50 micrometers are affected to create a porous, irregular surface. Subsequent infiltration by low viscosity resin polymerizing into the porosities, creating resin tags of various sizes, results in tremendous increase in surface area. The effect of enamel etching is demonstrated by outstanding clinical outcomes in non-carious cervical lesions without mechanical retention or enamel beveling when using three-step dentin adhesive systems[68].

2.3.1. Hydrophobic bonding

In contrast to glass-ionomer containing restorative materials, resin composites are generally hydrophobic in nature. This is because the chemical matrix is resin-based, including triethylene glycol dimethacrylate, urethane acrylate methacrylate, and bisphenol A-glycidyl methacrylate, which contain long chain hydrocarbons. This innate difference requires a conversion of the hydrophilic tooth substrate to a hydrophobic surface for interaction into the

resin matrix for dentin bonding, achieved through the use of dentinal bonding agents. Multiple varieties and generations of bonding agents provide the clinician with a wide variety of options, with generally superior performance among fourth generation etch-and-rinse and sixth generation two-step mild self-etching primer varieties[69]. Successful dentin bonding with these materials requires multiple clinical steps, including proper tooth preparation design, surface conditioning, primer and bond placements. Multiple steps provide more opportunities for error, leading to increased technique sensitivity. Unfortunately, attempts to decrease technique sensitivity for operator convenience using simplified dentin bonding protocols generally decreases clinical effectiveness compared to the gold standards previously mentioned[70].

2.3.2. Hydrophilic bonding

Glass-ionomer cements adhere directly to dental hard tissues through a mineral phase, evidenced by extensive investigation of direct chemical bonds between anionic polycarboxylic acid groups and cationic calcium ions, as well as hydrogen bonding[71]. As a result of carboxylic acid groups and hydroxyapatite interaction as observed by infrared spectroscopy, polyacrylate ions attach to minerals within the hydroxyapatite crystals by displacing phosphate ions[72].

Hydrophilic bonding potential lends glass-ionomers to the concept of minimally invasive dentistry[73]. Both glass-ionomers and resin-modified glass-ionomers are less prone to moisture sensitivity during restoration placement due to their hydrophilic nature and naturally bond to tooth structure[74].

2.3.3. Surface conditioning

The optimal surface conditioning process facilitates glass-ionomer molecules to establish both micro-mechanical and chemical bonds to the tooth surface[75]. Treating the prepared tooth surface with polyacrylic acids cleanses the tooth surface by removing the smear layer and

exposing collagen fibrils up to a micrometer deep[76]. The smear layer is a 0.5-5 micrometer conglomeration of debris created when teeth are surgically prepared[77]. Removal of the smear layer, tooth surface alterations and adhesion of glass ionomer cements to dentin depend on the duration, type and concentration of acid and resultant formation of intermediate layers containing metallic salts[78].

Improving the adhesion of glass-ionomers to enamel and dentin substrates is best achieved with high molecular weight acids with multiple functional groups; low molecular weight acids undesirably dissolve mineral content and degrade chemical bonding potential[79]. The typical surface conditioning concentration employed is 10-20% polyacrylic acid for ten seconds but may slightly vary by manufacturer[80]. This is in contrast to dentin bonding with resin composites, where micromechanical attachment to enamel and conversion of the hydrophilic organic components of dentin to hydrophobic surfaces is the overall goal.

2.3.3. Bond strength

Bond strength tests are generally used to evaluate dentinal bonding agents, however the same techniques can be used to evaluate glass-ionomer containing restorative material adhesion to other substrates. Modern techniques include micro-tensile bond strength and shear bond strength tests. Typically, specimens are prepared, aged in a medium or fatigued and then submitted to load or tension until fracture; subsequent evaluation of the fracture sites under magnification reveals mode of failure in the substrate, at the adhesive joint, or in the restorative material[81]. When the majority of the fractures occur at the adhesive interface, the adhesive bond strength is identified. Otherwise, the yield strength of either adherent or adherend is typically measured.

Many attempts at identifying the true bond strength of glass-ionomers to dentin ultimately result in measuring the yield or tensile strength of the glass-ionomer due to preponderance of fractures in the glass-ionomer[82]. Until the mechanical properties of the glass-ionomer improve, the true bond strength value to dentin will remain unknown.

2.3.3.1. Adhesion to other dental materials

Glass-ionomers adhere not only to tooth structure, but also other dental materials. Chemical bonding to surface oxide layers of precious metals has been demonstrated[83]. The tensile bond strengths between glass-ionomer cements and composite resins following acid etching are strong, resulting in cohesive fractures of the glass-ionomer[84]. If considering the adhesion to resin composite restorations, resin-modified glass-ionomers contain hydroxyethylmethacrylate, which readily lends a potential for adhesion with dentin bonding agents.

2.4. Physical and mechanical properties

In vitro evaluation of physical and mechanical properties may provide a basis for comparison of dental materials intended for similar functions, such as direct restorations. It is generally established that these tests, including flexural strength, compressive strength, coefficient of thermal expansion, etc. have some relevance to clinical performance. For example, identifying properties vastly inferior to materials with proven track records of success could at worst provide evidence for premature clinical failure and at best shorten the time required for clinical testing.

2.4.1. Flexural strength

Flexural strength is a measure of the force applied at fracture of a specimen, typically describing the ability of a material to resist deformation and fracture under load. In general,

glass-ionomers are the weakest compared to resin-modified glass-ionomers, which are in turn lower than resin composites[85]. High flexural strength is desired in restorative dental materials. An International Standards Organization specification describes a standard mechanism of measuring this mechanical property[86]. Some evidence suggests a correlation between abrasive wear and flexural strength in resin composite restorations[87].

2.4.2. Compressive strength

A mechanical property of restorative dental materials, this measurement is calculated by determining a failure load applied to a specimen's cross-section area. In general, an extensive study comparing the compressive strength, fluoride release and recharge of fluoride-releasing materials for glass-ionomer containing restorative materials revealed a negative linear correlation between the compressive strength and fluoride release, suggesting that restorative materials with a high fluoride release have lower mechanical properties[88].

2.4.3. Coefficient of thermal expansion

Restorative dental materials are affected by changes in temperature. The most significant effect as a result of temperature is a change in volume. This property is quantified by the coefficient of thermal expansion, measured as a rate by which the material expands in length for each degree increase in temperature. The coefficient of thermal expansion for type 2 glass-ionomers are reportedly the most similar to tooth enamel of all dental materials[89]. This property may play a role in maintaining marginal seal, as the restorative material and tooth expand and contract at similar rates[90].

2.4.4. Bioactive availability

Glass-ionomers have the potential for ion exchange with the tooth substrate and oral environment, a physical property recently re-branded as "bioactivity". Although limited potential

benefits may be available from calcium release, the potentially most significant ion is fluoride due to its multiple modes of caries suppression. However, there is no proven fluoride concentration to establish caries inhibition[91]. Evidence is available to demonstrate tooth structure can be influenced by adjacent fluoride containing bioactive restorative materials[92].

2.4.4.1 Cumulative fluoride release

The glass-ionomer matrix is a hydrated polygel, enabling continuous ion exchange in a fluid environment. These fluoride ions can be released from the material, but the rate decreases in concentration over time as the potential is exhausted[93].

2.4.4.2. Fluoride re-uptake

Not only can fluoride be released from glass-ionomer containing restorative materials, but the same fluoride ions can be re-established withing the polygel matrix from sources such as fluoridated toothpastes or varnishes. As a result, the material can serve as a reservoir for fluoride[94,95].

2.4.4.3. Effect of coating on fluoride availability

Coatings placed on the surface of glass-ionomer containing restorative materials reduces the total volume of fluoride release, likely based on reduced exposed surface area available for ion exchange with the oral environment[96]. Currently available nano-filled resin coatings are applied in a single coat are approximately five to ten micrometers thick[97].

2.5. Fatigue

Dental materials experience thermal challenges, changes in pH and forces of mastication in function. Combined, these environmental challenges stress the material over time. The goal of any dental restorative material should be to withstand these challenges for a long period of

clinical service. Conventional glass-ionomers are exceptionally susceptible to fatigue, leading to early clinical failures due to unacceptable wear rates.

The addition of fatiguing specimens within in vitro studies measuring physical and mechanical properties is generally encouraged to reflect behaviors more likely to occur in a clinical environment[98]. Fatigue is generally the combination of repeated mechanical loading and a combination of thermocycling. Mechanical loading stresses the material under controllable settings, such as force, duration and frequency. Bite forces can exceed 150 N when the muscles of mastication are maximally exerted[99]. However, the average biting force is estimated at 49 N[100], with forces on posterior teeth exceeding 100 N at rates approximately 1.5 Hz in the wet oral cavity[101]. Thermocycling is a means to artificially age a dental material[102]. A chewing simulator is a method to achieve both means of fatigue simultaneously.

2.5.1 Wear

There are many definitions of wear, but a basic definition describes multifactorial processes leading to the loss of dental hard tissues. In dentistry, relatively high wear rates are generally undesirable in restorations as loss of anatomic contour may result in unfavorable masticatory force distribution leading to fracture or loss of surface finish diminishing esthetics[103]. For many years, the relatively high wear rates of resin composites in permanent posterior teeth in comparison to amalgam was a genuine concern[104]. In relation to natural tissues, tooth enamel wears less, approximately 20 micrometers annually[105]. As such, defined limits of linear wear tolerance were set forth for resin composites in posterior teeth. Specifications for acceptable wear rates in the 1980s and 1990s required no more than 250 micrometers of vertical material loss over a four year period; in 2003, posterior composites in stress-bearing restorations was still a controversial issue to the point that even those materials deemed “acceptable” with the

American Dental Association seal of approval "should not be used for large stress bearing restorations" [106]. Fulfilling these clinical observations required at minimum two clinical studies of at least 18 months in duration prior to consideration for earning the seal of acceptance.

Fig 2. American Dental Association seal of acceptance annotating that accepted resin composites were still not indicated for large stress bearing restorations as recent as 2003.

The requirements set forth by the American Dental Association continued to change over time for resin composites, evolving to clinical evidence of surface wear not exceeding 50 micrometers over an 18-month period[107]. The seal of acceptance program for professional products was discontinued in 2007. Conventional glass-ionomer cements historically wear faster compared to resin composites by a factor of up to three times[108].

In vitro wear comparisons of conventional glass-ionomers, resin-modified glass-ionomers, metal-reinforced glass-ionomers, resin composites and amalgam revealed the early generations of glass-ionomers exhibited significantly higher wear rates than dental amalgam[109]. Corresponding wear specifications for glass-ionomers similar to resin composites were never implemented; glass-ionomer containing restorative material mechanical properties were consistently inferior to amalgams and resin composites and thus were never indicated for use as

posterior, load-bearing restorations. Regardless of current advertising tactics of load-bearing glass-ionomer containing restorative materials, specifications or requirements for wear to date do not exist for neither resin composites nor glass ionomer containing restorative materials[110].

There are four basic mechanisms by which surfaces can wear, including abrasive, fatigue, corrosive and adhesive[111]. Direct restorative dental materials are most susceptible to abrasive and fatigue wear and heavily influenced by individual chemical composition[112].

2.5.1.1 Abrasive wear

Abrasive wear is a frequent mechanism in dentistry and is composed of two- and three-body wear[113]. In the former, a relatively rough surface with protuberances scrapes off an opposing surface in motion. In the latter, particles are caught between an interface and contribute to loss of structure on one or both opposing surfaces. Clinical examples of each include wear of natural tooth structure opposing a rough feldspathic crown (two-body wear) and toothpaste abrasion (three-body wear)[114]. This category of wear is significant in dentistry, as dentifrices must demonstrate performance on a scale of relative dentin abrasion (RDA) under 250 to achieve Federal Drug Administration approval for market release[115].

2.5.1.2. Fatigue wear

Fatigue wear is caused by repeated material stressing which over time creates subsequently larger cracks within the material[116]. After continued stress in this regard, crack propagation and accumulation reach a critical moment at which point a larger piece fractures away from the surface. This phenomenon occurs in both the natural dentition and restorative materials. Materials with internal voids or irregularities present an inherent risk of failure due to fatigue wear because cracks rapidly expand across these weak points, which is a concern with a

high percentage of air trapped within glass-ionomer polygel matrices as a result of mixing processes[117].

2.5.1.3. Corrosive wear

Corrosive wear, or more commonly referred to as erosive wear in dentistry, involves the loss of surface structure following an acidic challenge of non-cariogenic origin. This process is also referred to as erosive tooth wear[118]. The diet can be a major contributory factor[119] as well as the presence of saliva[120]. In general, dental materials outperform the natural dentition, are not susceptible to corrosive wear, and this process is less significant compared to abrasive and fatigue wear[121].

2.5.1.4 Adhesive wear

A relatively rare mechanism in dentistry in which an antagonist bonds more tightly under compression to a surface resulting in wear describes adhesive wear[122]. This phenomenon is likely minimized due to saliva acting as a lubricating layer in the mouth[123]. However, the bond strength between hydroxyapatite and glass-ionomer containing restorative materials typically reveal cohesive fracture within the material, suggesting the bond to tooth structure is greater than the inherent tensile strength. In the event glass-ionomer containing restorative materials are used as load-bearing restorations, the potential for this mechanism of wear should not be ignored as a potential contributing factor in overall wear performance.

2.5.2. In vitro simulation

The International Organization for Standardization describes a variety of testing methods for wear[124]. However, a tremendous variation in testing parameters makes comparison of in vitro study results largely difficult to interpret with no clear correlation to clinical performance demonstrated; however, evaluating new material concepts, systems or technologies should

involve laboratory wear evaluations before the materials are released for clinical trials[125]. Another advantage to in vitro simulation is the efficient facilitation of wear measurement using digital profilometry, as the smallest clinically discernable step is approximately 100 micrometers. There is not consensus on in vitro or chewing simulator parameters.

2.5.2.1 Thermocycling

In vitro fatigue testing typically involves thermocycling (thermal cycling). Mechanical stresses induced by volumetric changes occur within a material then the temperature rises and falls[127]. Intervals of 10,000 thermocycles approximates one year of artificial aging.

2.5.2.2. Correlation to clinical performance

There is not yet a simulation methodology that fully replicates the oral environment, but a long-standing goal has been to create a method to directly correlate in vitro to clinical wear[128] (). It is proposed that 200,000-400,000 in vitro chewing cycles might approximate one year of clinical service depending on the chewing simulator[129,130,131]. Although still in development, a method to correlate and simulate clinical performance would be an outstanding advancement in dental materials science, as the vast majority of clinical trials for direct restorative materials science are less than five years in length and establish safety and efficacy in lieu of meaningful predictions untoward long term performance[132].

CHAPTER 2: RESEARCH STUDY

1. Introduction

This study compared in vitro wear of contemporary glass-ionomer containing dental materials commercially advertised for use in the permanent dentition as load-bearing posterior restorations.

2. Specific aims of thesis:

Specific aim: Quantify and compare the volumetric wear of three glass-ionomer containing restorative materials and a resin composite.

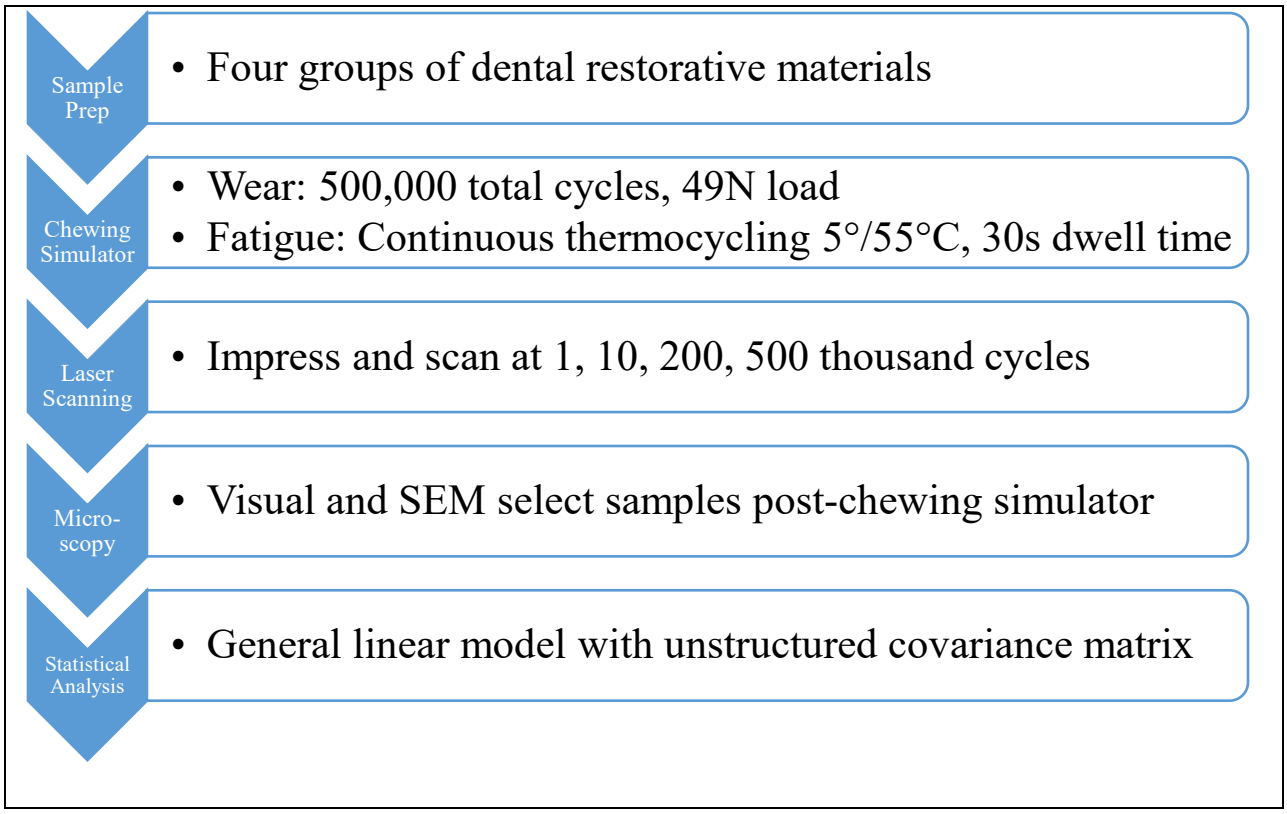
3. The null hypothesis:

Wear volumes of contemporary glass-ionomer containing materials advertised for use in posterior dentition as load-bearing posterior restorations have no difference compared to a contemporary resin composite.

4. Materials and methods:

Four restorative dental materials were used in this study. Ionolux (VOCO America Inc.) is a resin-modified glass-ionomer. Activa Bioactive Restorative (PULPDENT) is a bioactive ionic resin with reactive glass filler. Equia Forte HT and Equia Coat (GC America Inc.) is a high viscosity glass-ionomer hybrid system. Filtek Supreme Ultra (3M ESPE) is a visible light-activated resin composite.

Fig 3. Method summary flowchart.



4.1. Material used

Resin composite and glass-ionomer containing restorative materials are used in this study (Table 3.).

Table 3. Materials used in this study according to respective manufacturers.

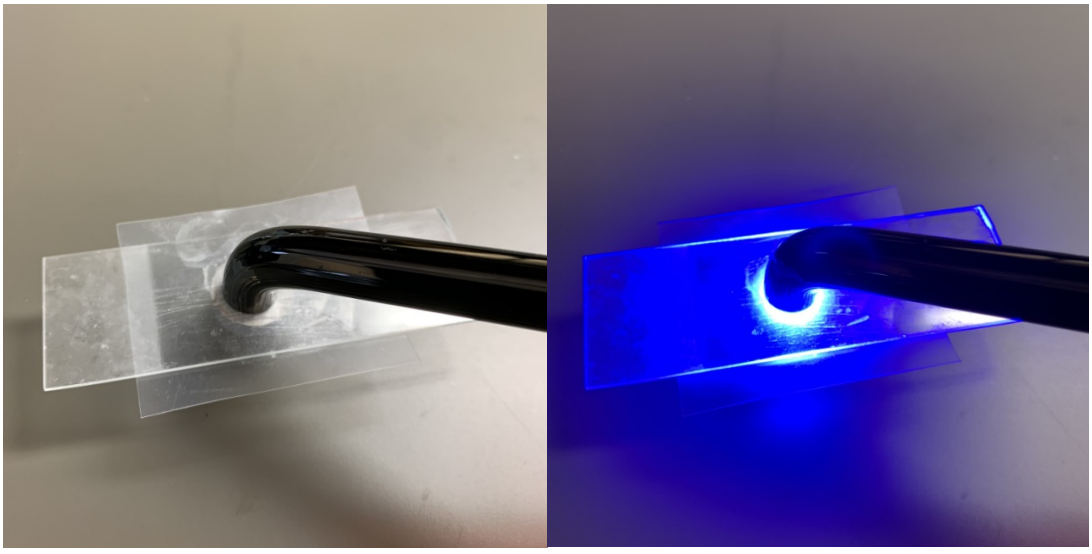
Product	Description
Filtek Supreme Ultra	Visible light-activated composite
Equia Forte HT & Equia Coat	High viscosity glass-ionomer hybrid glass system
Ionolux	Resin-modified glass-ionomer restorative
Activa Bioactive Restorative	Bioactive ionic resin with reactive glass filler

4.2. Specimen preparation

Standardized flat cylindrical disc specimens of each material were prepared. Equia Forte HT and Ionolux material capsules were activated, mixed for ten seconds at 4,000 oscillations per

minute in an amalgamator (KERR Automix, USA) and placed directly into specimen crucibles. Equia Coat resin was applied immediately to Equia Forte HT by dispensing the liquid into a dappen dish and thoroughly brushing onto the Equia Forte HT surface. Filtek Supreme Ultra and Activa Bioactive Restorative specimens were prepared by dispensing material from original carpules or automix syringes directly into the specimen crucibles in increments not exceeding manufacturer recommendations. After dispensing the final layer, all specimens were expediently covered with a mylar strip and flat glass slide and polymerized for 20 seconds with a curing light (Elipar DeepCure-S LED Curing Light, 3M, USA). The specimens were wet polished using 600 and 1200 grit sandpaper (CarbiMet, Buehler, USA) and placed into a distilled water ultrasonic bath for five minutes to remove polishing debris. Prepared specimens were placed in deionized water at 37 degrees Celsius for 24 hours prior to mounting in the chewing simulator for fatiguing protocol.

Fig 4. Preparing and photopolymerizing specimens directly within specimen holders.

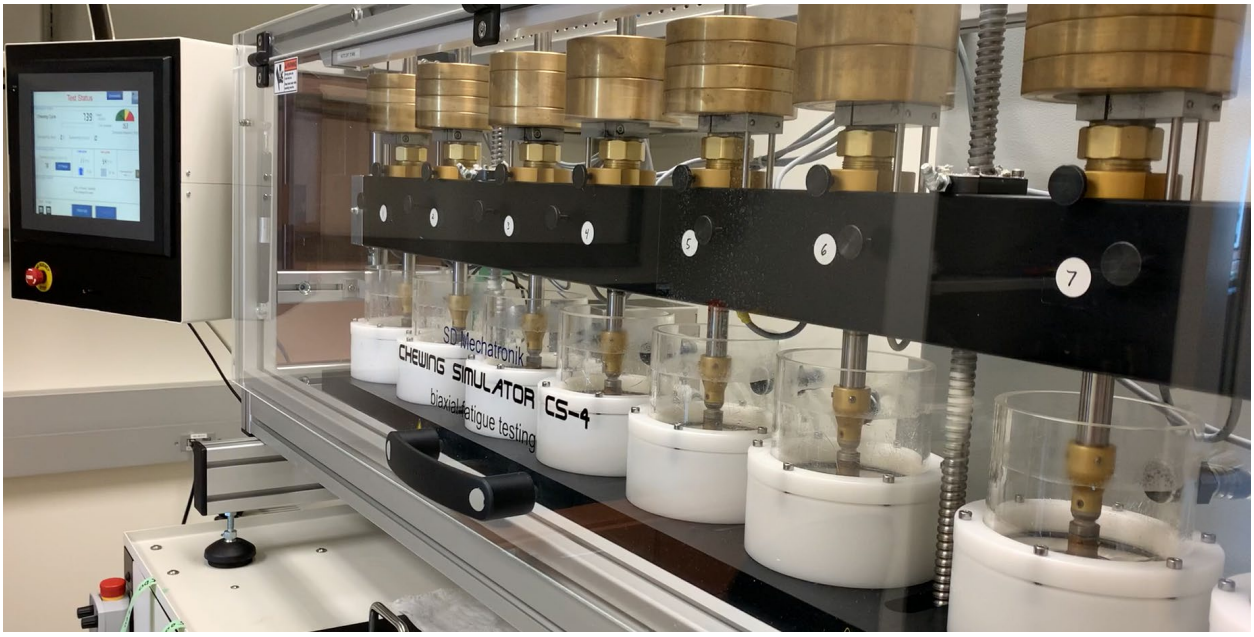


4.3. Fatiguing protocol

Specimens were fatigued in an eight-bay chewing simulator (CS-4, SD Mechatronik, Germany). Continuous interval loading was undertaken using a six millimeter steatite ball antagonist striking the specimens at a 90 degree angle from a two millimeter height with a 60 millimeter per second speed and a vertical loading force of 49N followed by an immediate 0.7 millimeter horizontal slide stroke with an overall frequency of 1.6Hz (96 beats per minute). The horizontal back-forth motion occurred at a 40 millimeter per second rate. Simultaneously, the mounted specimens were subjected to continuous liquid thermal cycle bathing under distilled water at alternating temperatures of 5 and 55 degrees Celsius for an average total of 4546 cycles per group. Temperatures were maintained for 30 second dwell times at each thermal cycle interval. A total number of 500,000 mechanical loading cycles were achieved per specimen.

Fig 5. Steatite antagonist mounted in polymethylmethacrylate.

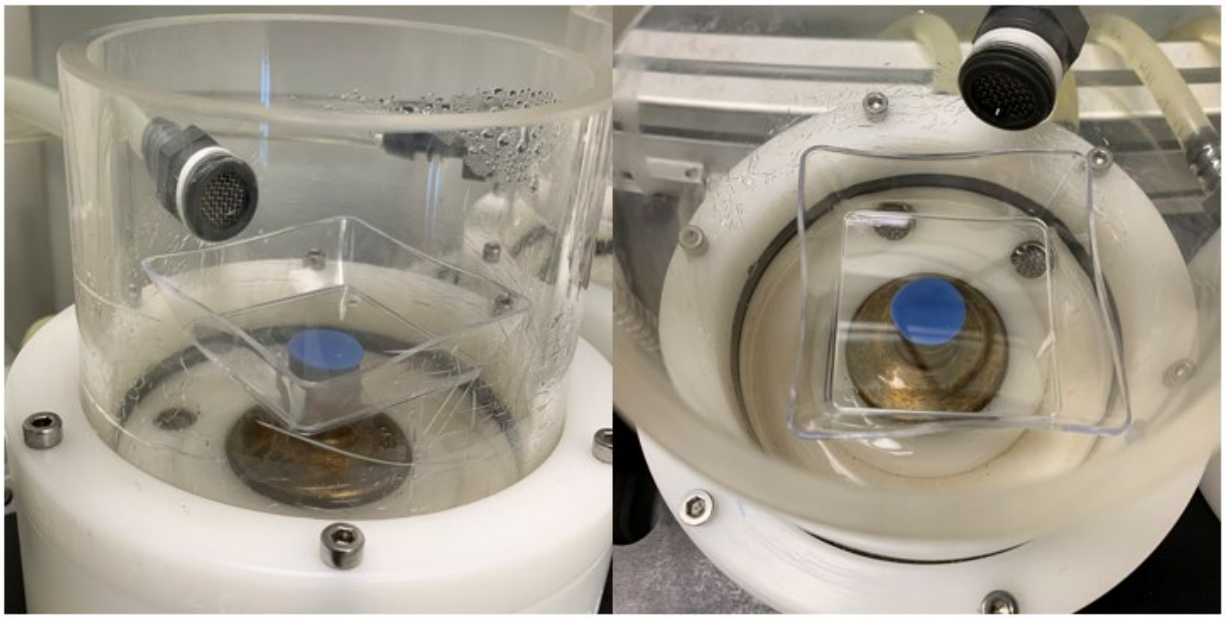
Fig 6. SD Mechatronik chewing simulator with specimens under load.



4.4. Laser scanning

Analog impressions were made of mounted samples during the fatiguing protocol at prescribed intervals (1,000, 10,000, 200,000. 500,000 loading cycles) using low viscosity polyvinyl siloxane impression material (Extrude Type 3: Low Consistency, Kerr, USA). To facilitate making the impression without disturbing the specimens, the chewing simulator was paused and the specimens were dried with compressed air immediately prior to making the impressions (Dust-Off, Falcon Safety Products, Inc., USA). A flat plastic surface aided in compressing the impression material onto the specimens. The self-cured impression material was removed after ten minutes and fatiguing protocol resumed.

Fig 7. Impression-making of the wear facet in specimens at prescribed intervals.



The impressions of the wear facet were subsequently scanned in a laser scanner (Laserscanner LAS-20, SD Mechatronik, Germany) with up to 40 micrometer resolution and analyzed using computer software (Geomagic, Germany) to digitally calculate volume against a flat plane using three points of reference.

Fig 8. Laserscanner LAS-20.

Fig 9. LAS-20 software with impression of specimen ready to scan.

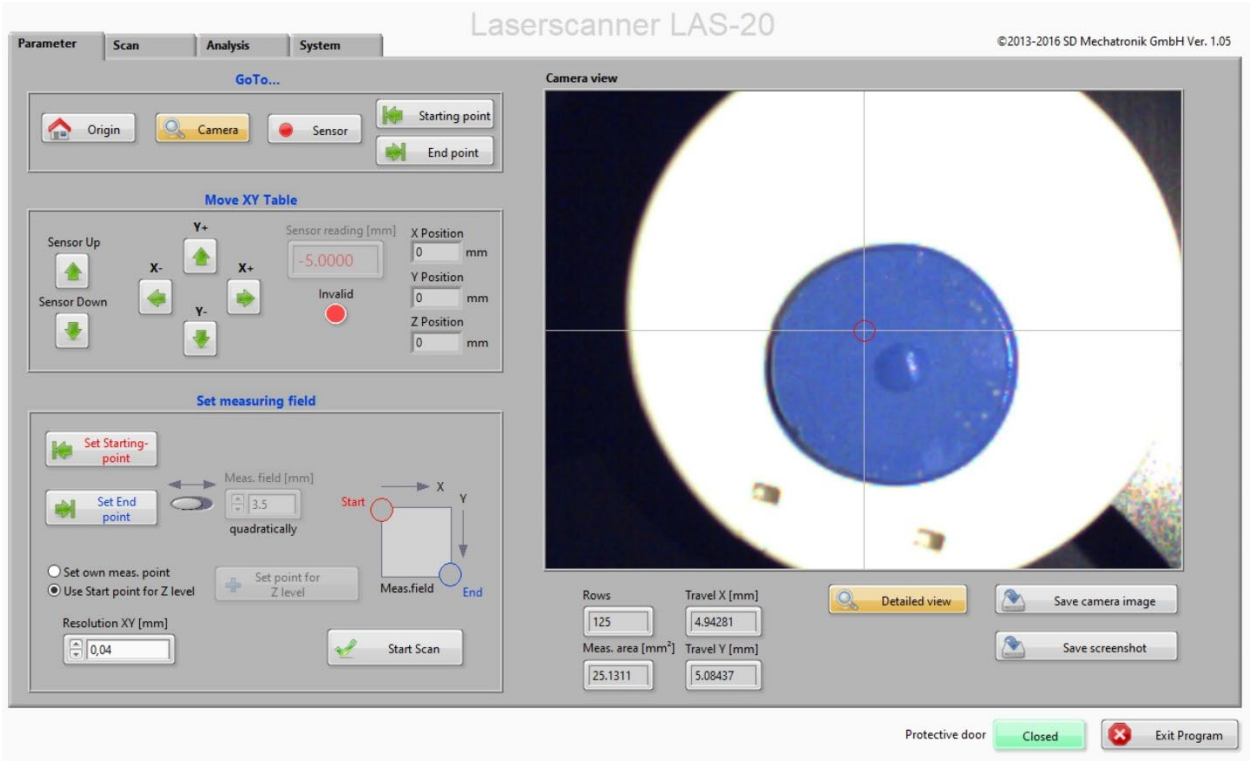


Fig 10. LAS-20 scan of impression of specimen wear facet at 500,000 cycles.

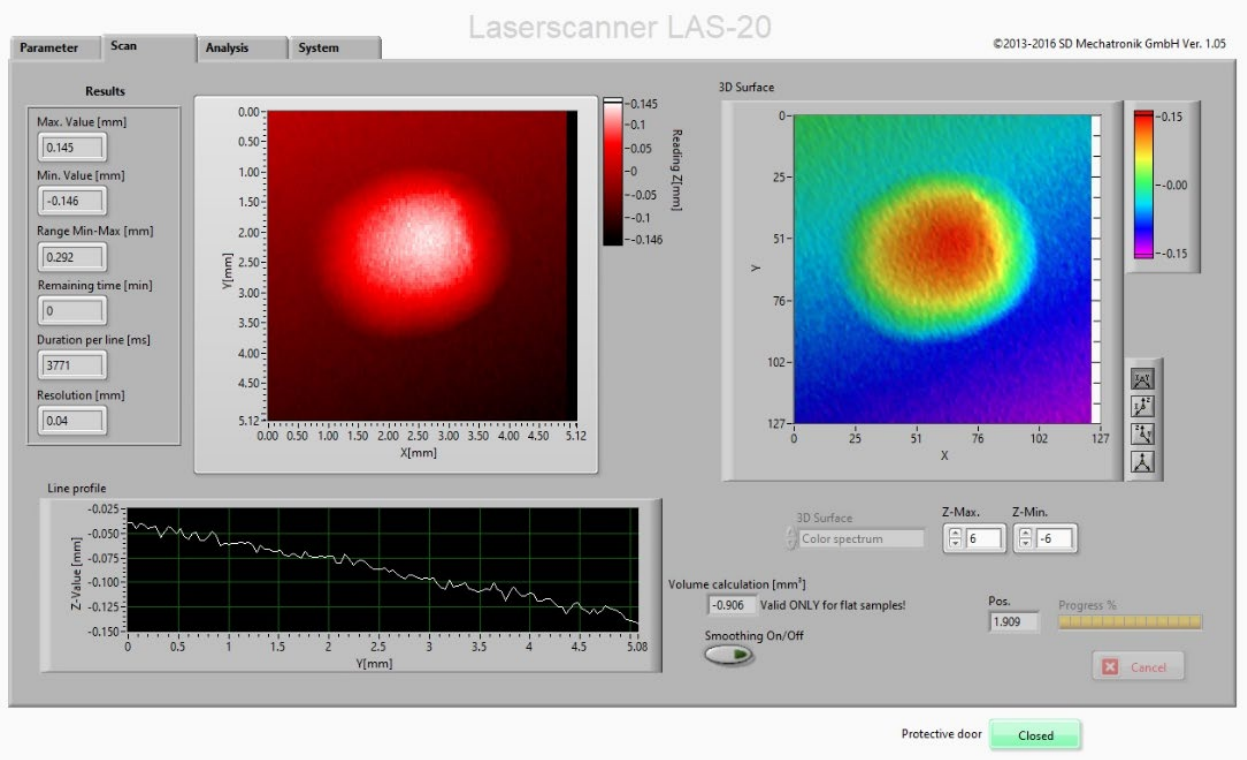
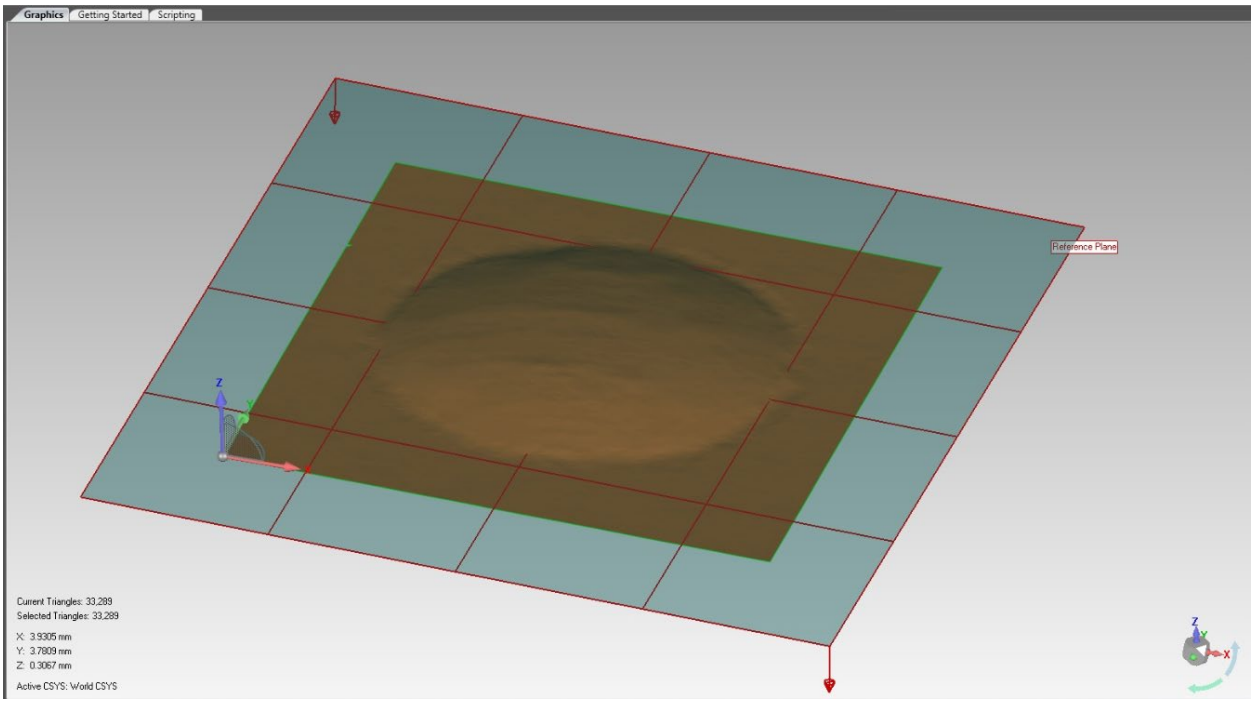


Fig 11. Software computing volume of wear facet referencing a three-point plane.



4.5. Microscopy

Upon completion of fatiguing protocol, select samples of Equia Forte HT were submitted for SEM evaluation for visualization. Light microscopy up to 40x was used to visualize select samples.

5. Statistical analysis

The data was fit to a general linear regression model. The model assumed an unstructured covariance matrix for each subject with the same covariance matrix for all subjects regardless of assignment with 44 degrees of freedom. A Bonferroni adjustment for multiple comparisons was performed for all tests in the differences of least squares means analysis. The adjusted alpha was 0.0021 and adjusted confidence intervals were 99.8%. For the final 500,000 interval, a one-way analysis of variance was used to assess the differences in mean as a result of the chewing simulator among the materials: glass-ionomer containing restorative materials (Activa Bioactive Restorative, Equia Forte HT and Ionolux) and a resin composite control (Filtek Supreme Ultra). LSMEANS function in SAS v 9.4 Proc GLM was used to compare the means for all possible pairs of materials. The level of significance was set at 0.05.

6. Results

Fig 12. Activa Bioactive Restorative wear facet following 500,000 cycles. Divider = 1mm.

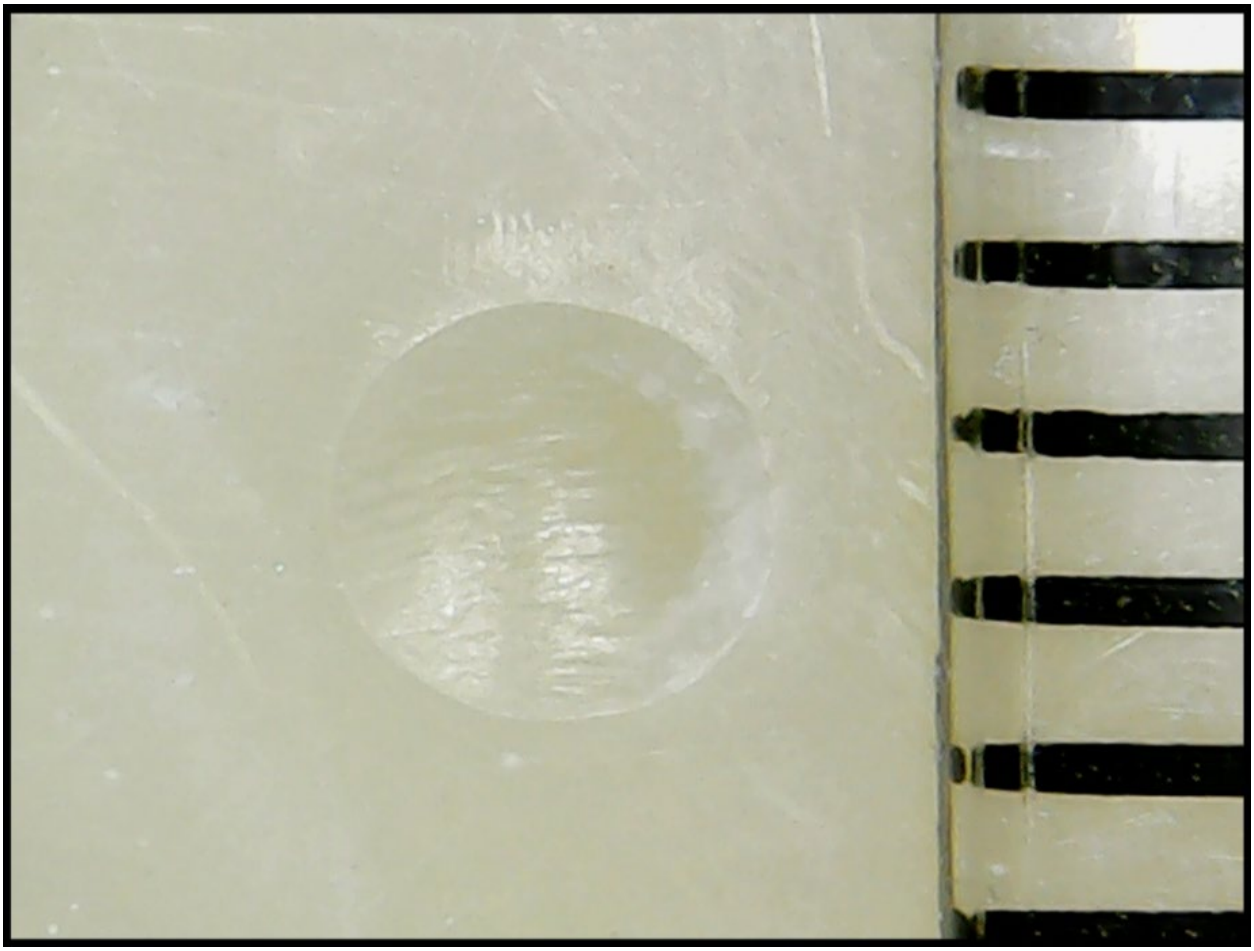


Fig 13. Equia Forte HT wear facet following 500,000 cycles. Divider = 1mm.

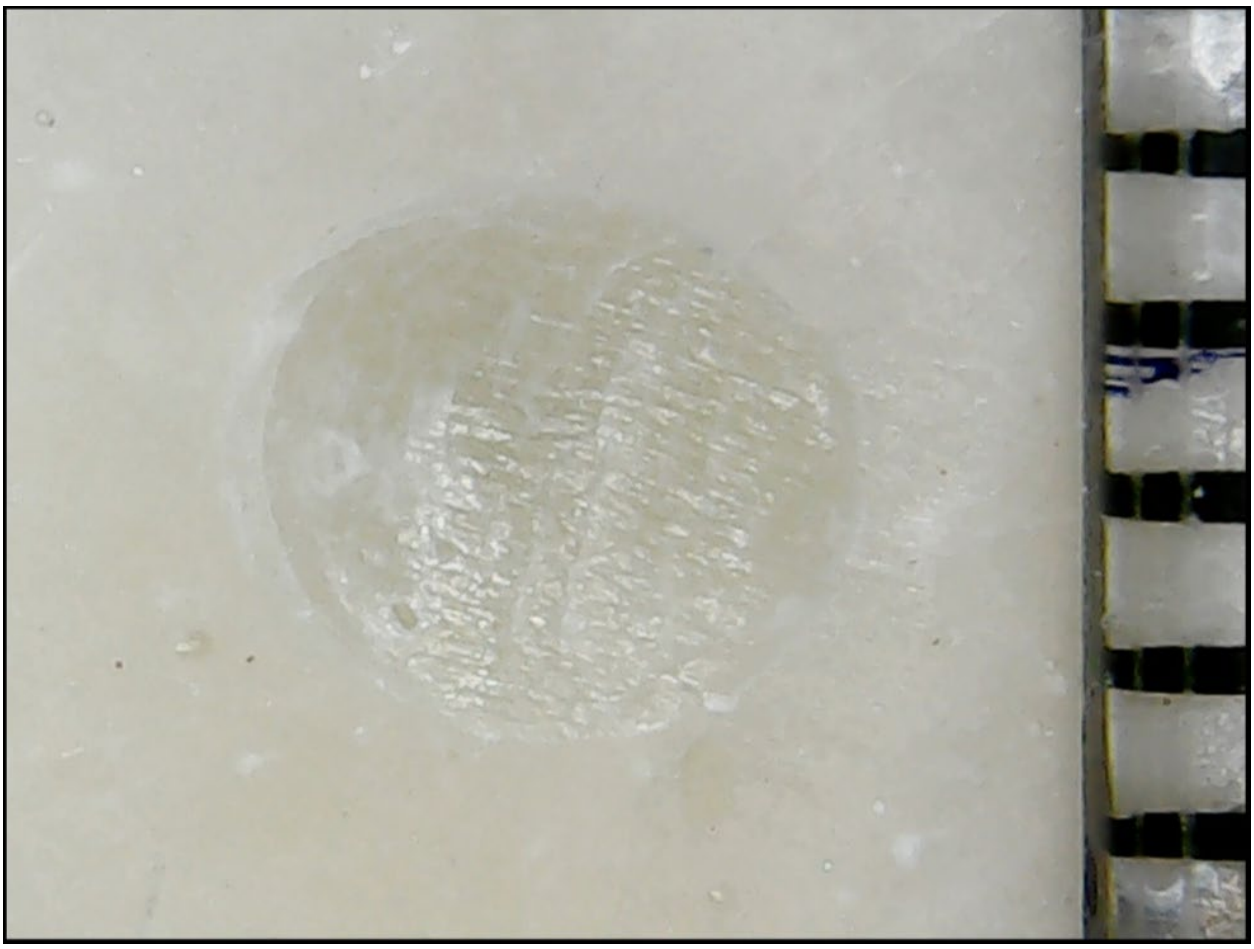


Fig 14. Ionolux wear facet following 500,000 cycles.

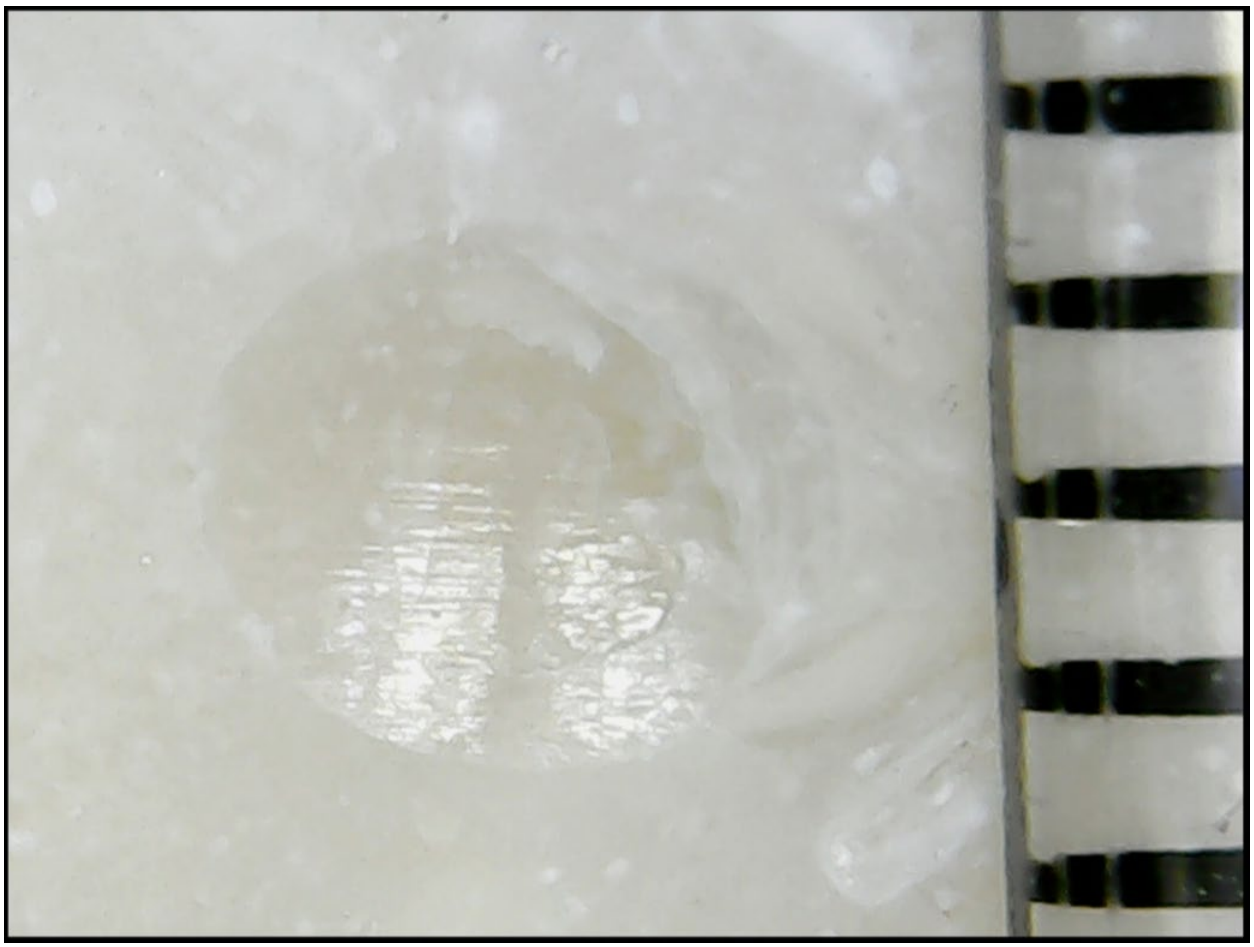


Fig 15. Equia specimen 45 degree tilt SEM following 500,000 cycles. The specimen was sectioned across the wear facet.

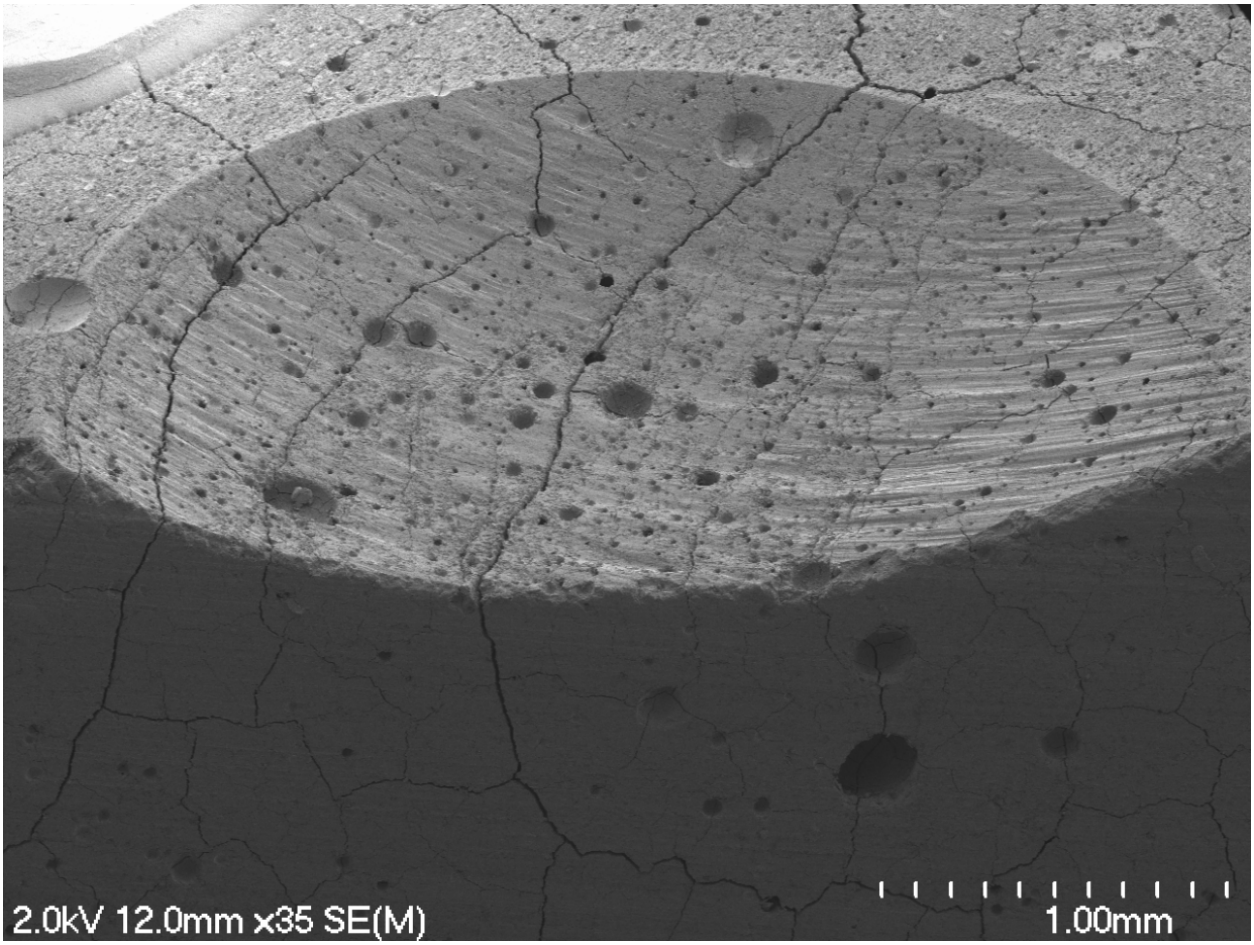


Fig 16. Equia specimen facet profile SEM following 500,000 cycles.

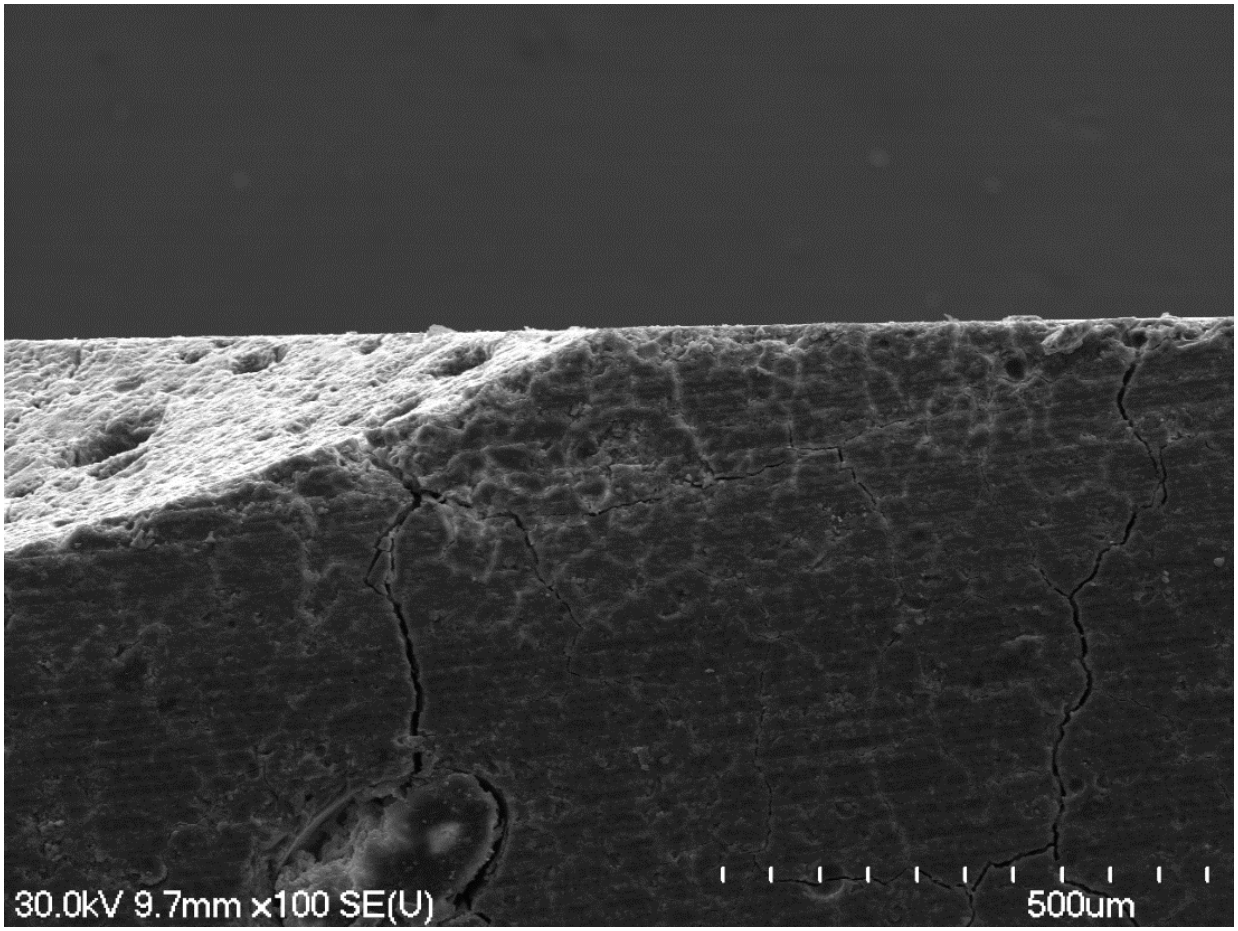
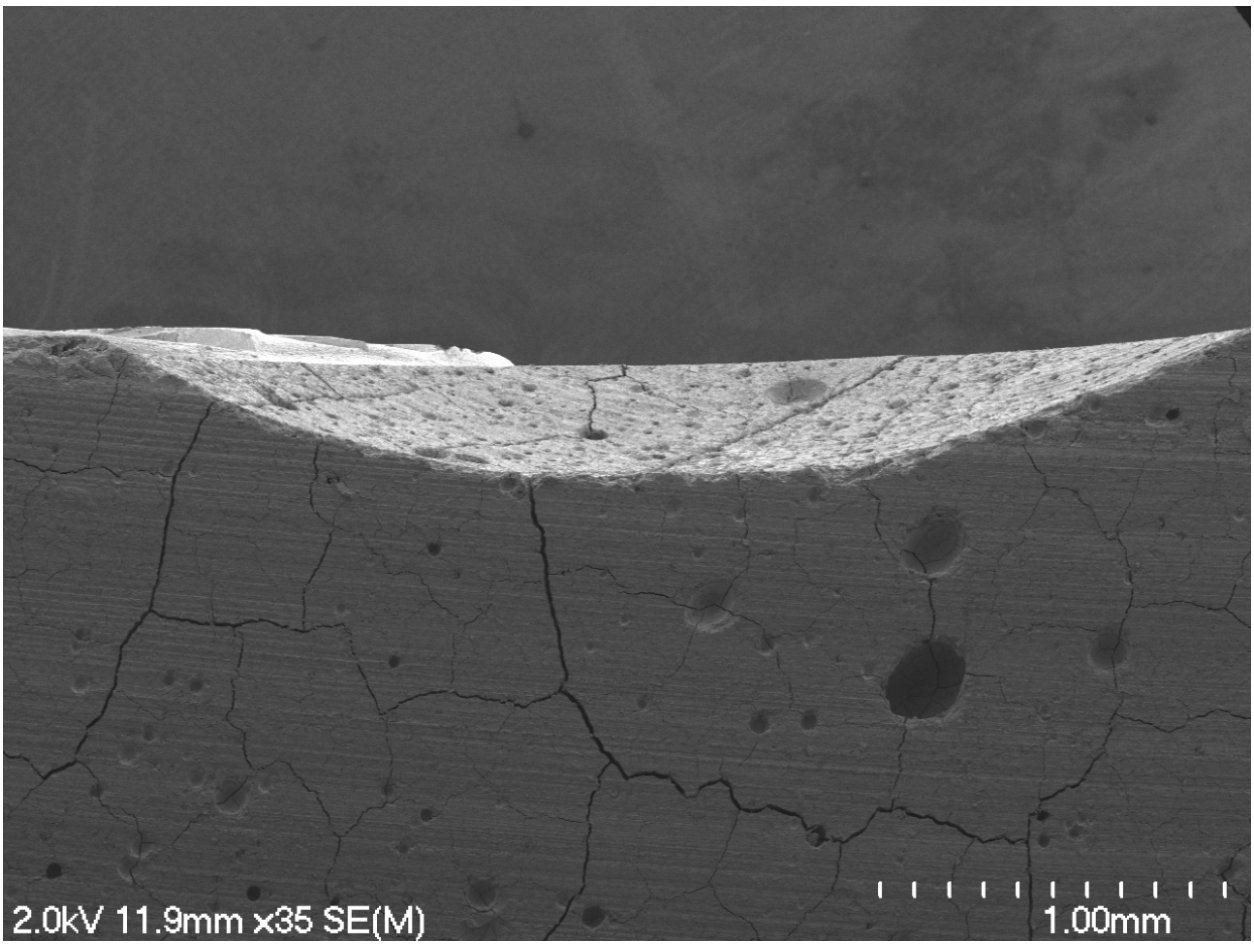


Fig 17. Equia specimen 90 degree tilt SEM following 500,000 cycles.



6.1. Volumetric loss due to wear

Resulting measurements of cumulative volumetric loss of material due to chewing simulator wear are reported in table 4.

Table 4. Cumulative volumetric loss of material due to chewing simulator wear.

Sample	Interval (thousand cycles) (cubic millimeters)				
	1	10	200	500	
Filtek 1	0.002698	0.046038	0.384411		
Filtek 2	0.003813	0.031884	0.451920		
Filtek 3	0.002580	0.045035	0.446486	0.885034	
Filtek 4	0.004486	0.034917	0.421776	0.896892	
Filtek 5	0.005166	0.040250	0.390158	0.754561	
Filtek 6	0.002542	0.044491	0.455343	0.909574	
Filtek 7	0.003138	0.045415	0.341083	0.636837	
Filtek 8	0.004859	0.039754	0.420552	0.849586	
Filtek 9	0.006664	0.036003	0.376757	0.696748	
Filtek 10	0.003007	0.032354	0.394068	0.677303	
Filtek 11	0.002058	0.033451	0.351006	0.724362	
Filtek 12	0.004725	0.042692	0.399727	0.772372	
Activa 1	0.005886	0.022436	0.288610		
Activa 2	0.005188	0.025972	0.284508		
Activa 3	0.005604	0.028205	0.248243	0.499570	
Activa 4	0.005911	0.032162	0.234100	0.559765	
Activa 5	0.003413	0.018253	0.220590	0.576246	
Activa 6	0.004657	0.020125	0.224599	0.464627	
Activa 7	0.004856	0.019735	0.201086	0.477909	
Activa 8	0.004393	0.022742	0.220997	0.394173	
Activa 9	0.004285	0.030211	0.199881	0.376846	
Activa 10	0.004719	0.019231	0.217867	0.401457	
Activa 11	0.005959	0.019558	0.192821	0.368758	
Activa 12	0.006482	0.019901	0.197198	0.358346	
Ionolux 1	0.028852	0.094692	0.653021		
Ionolux 2	0.018562	0.118824	0.571981		
Ionolux 3	0.053324	0.116388	0.503342	1.066634	
Ionolux 4	0.164669	0.279546	0.654232	0.993507	
Ionolux 5	0.063346	0.202529	0.561441	0.773917	
Ionolux 6	0.097895	0.163650	0.621876	0.836529	

Ionolux 7	0.056687	0.231831	0.649462	1.053670
Ionolux 8	0.042881	0.213892	0.625207	1.048803
Ionolux 9	0.072058	0.177950	0.360620	0.519702
Ionolux 10	0.053411	0.102395	0.610371	0.778729
Ionolux 11	0.058215	0.105387	0.465685	0.612565
Ionolux 12	0.036953	0.118255	0.411382	0.637022
Equia 1	0.163171	0.272087	0.947710	
Equia 2	0.020892	0.572535	1.375913	
Equia 3	0.160673	0.302966	1.215979	2.257207
Equia 4	0.041227	0.358391	1.213867	1.760675
Equia 5	0.136782	0.291773	0.997601	1.217329
Equia 6	0.190658	0.323525	1.488232	2.252012
Equia 7	0.111508	0.214591	0.841813	1.560962
Equia 8	0.163901	0.299760	1.280752	1.775962
Equia 9	0.174892	0.244775	0.546258	0.836321
Equia 10	0.228752	0.374592	0.802458	1.059116
Equia 11	0.193818	0.277361	0.883969	1.283607
Equia 12	0.160075	0.349108	0.878936	1.490594

Mean	Interval				
	1	10	200	500	
Filtek	0.003811	0.039357	0.402774	0.780327	(cubic millimeters)
Activa	0.005113	0.023211	0.227542	0.44777	
Ionolux	0.062238	0.160445	0.557385	0.832108	
Equia	0.145529	0.323455	1.039457	1.549379	

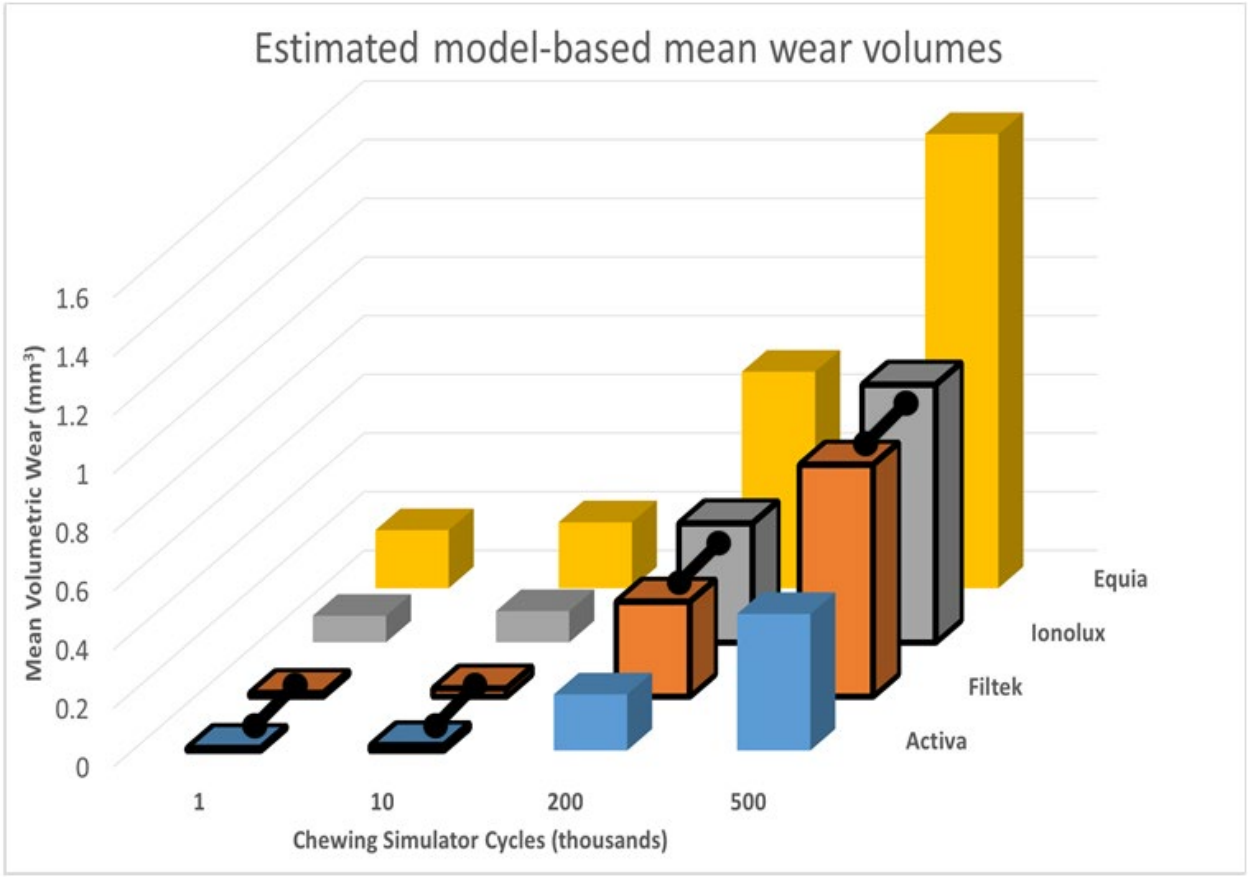
Standard Deviation					
	Interval				
Filtek	1	10	200	500	(cubic millimeters)
Activa	0.000884	0.004756	0.031953	0.07973	
Ionolux	0.03826	0.060606	0.100074	0.201993	
Equia	0.060972	0.091117	0.275185	0.474223	

Differences of Least Squares Means											
Effect	**Material**	**Material**	**W_Ind**	**Estimate**	**Standard Error**	**DF**	**t Value**	**Pr > |t|**	**Alpha**	**Lower**	**Upper**
Material	Activa	Equia	0.00	-0.1892	0.01087	44	-17.41	<.0001	0.0021	-0.2248	-0.1537
Material	Activa	Filtek	0.00	-0.00296	0.01087	44	-0.27	0.7866	0.0021	-0.03852	0.03260
Material	Activa	Ionolux	0.00	-0.08195	0.01087	44	-7.54	<.0001	0.0021	-0.1175	-0.04639
Material	Equia	Filtek	0.00	0.1863	0.01087	44	17.14	<.0001	0.0021	0.1507	0.2218
Material	Equia	Ionolux	0.00	0.1073	0.01087	44	9.87	<.0001	0.0021	0.07171	0.1428
Material	Filtek	Ionolux	0.00	-0.07899	0.01087	44	-7.27	<.0001	0.0021	-0.1146	-0.04343
Material	Activa	Equia	1.00	-0.2071	0.01065	44	-19.45	<.0001	0.0021	-0.2419	-0.1722
Material	Activa	Filtek	1.00	-0.00939	0.01065	44	-0.88	0.3829	0.0021	-0.04423	0.02546
Material	Activa	Ionolux	1.00	-0.08856	0.01065	44	-8.32	<.0001	0.0021	-0.1234	-0.05372
Material	Equia	Filtek	1.00	0.1977	0.01065	44	18.57	<.0001	0.0021	0.1629	0.2326
Material	Equia	Ionolux	1.00	0.1185	0.01065	44	11.13	<.0001	0.0021	0.08368	0.1534
Material	Filtek	Ionolux	1.00	-0.07918	0.01065	44	-7.43	<.0001	0.0021	-0.1140	-0.04433
Material	Activa	Equia	20.00	-0.5465	0.04263	44	-12.82	<.0001	0.0021	-0.6860	-0.4070
Material	Activa	Filtek	20.00	-0.1315	0.04263	44	-3.08	0.0035	0.0021	-0.2710	0.008022
Material	Activa	Ionolux	20.00	-0.2142	0.04263	44	-5.02	<.0001	0.0021	-0.3537	-0.07471
Material	Equia	Filtek	20.00	0.4150	0.04263	44	9.74	<.0001	0.0021	0.2755	0.5545
Material	Equia	Ionolux	20.00	0.3323	0.04263	44	7.79	<.0001	0.0021	0.1928	0.4718
Material	Filtek	Ionolux	20.00	-0.08273	0.04263	44	-1.94	0.0587	0.0021	-0.2222	0.05676
Material	Activa	Equia	50.00	-1.0824	0.1070	44	-10.12	<.0001	0.0021	-1.4324	-0.7323
Material	Activa	Filtek	50.00	-0.3242	0.1070	44	-3.03	0.0041	0.0021	-0.6743	0.02583
Material	Activa	Ionolux	50.00	-0.4126	0.1070	44	-3.86	0.0004	0.0021	-0.7626	-0.06251
Material	Equia	Filtek	50.00	0.7581	0.1070	44	7.09	<.0001	0.0021	0.4081	1.1082
Material	Equia	Ionolux	50.00	0.6698	0.1070	44	6.26	<.0001	0.0021	0.3197	1.0199
Material	Filtek	Ionolux	50.00	-0.08834	0.1070	44	-0.83	0.4134	0.0021	-0.4384	0.2617

Table 5. P-value calculation using SAS statistical software following 500,000 cycles.

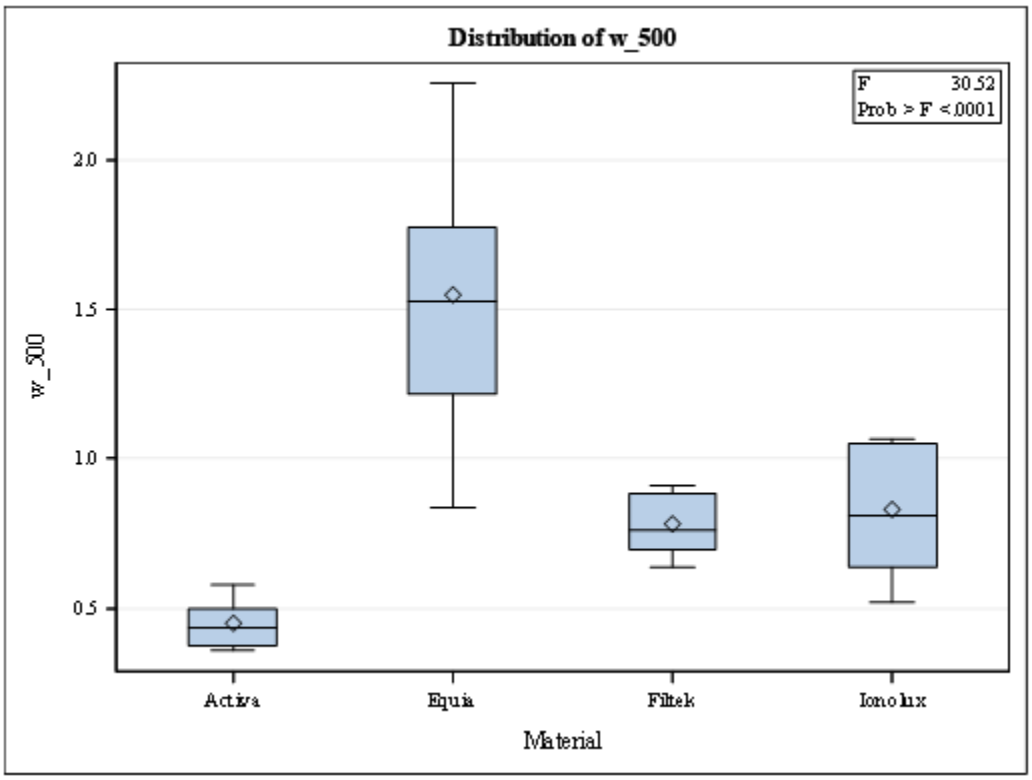
Least Squares Means for effect Material Pr > |t| for H0: LSMean(i)=LSMean(j) P-Value (alpha = 0.05)				
	Activa	**Equia**	**Filtek**	**Ionolux**
Activa				
Equia	<.0001			
Filtek	0.0081	<.0001		
Ionolux	0.0026	<.0001	0.6653	

Fig 17. General linear model assuming an unstructured covariance matrix for each subject assuming the same covariance matrix for all subjects regardless of assignment.



At the final 500,000 interval, there was a statistically significant difference in mean volumetric wear for Activa Bioactive Restorative (P=0.0081, 95% CI: 0.3973, 0.4982) and Equia Forte HT (P<0.001, 95% CI: 1.2495, 1.8493), but no statistically significant difference in mean volumetric wear for Ionolux (P=0.6653). Activa Bioactive Restorative wore approximately 60% less than, and Equia Forte HT twice more than Filtek Supreme Ultra on average, respectively.

Fig 19. General linear model assuming an unstructured covariance matrix.



7. Discussion

The aim of this study was to evaluate glass-ionomer containing restorative materials advertised for use as load-bearing restorations despite historical contraindications in this clinical application. The reasons include poor wear resistance and bulk fracture. Regardless of these known limitations, there are continuous efforts to place glass-ionomer containing restorative materials as load-bearing restorations based on a seemingly "easy-to-use" basis[133]. The materials in this study were selected because of manufacturer claim as suitability as load-bearing restorations in the posterior dentition. To evaluate wear, the in vitro wear produced by a chewing simulator was compared among three materials and a well-accepted resin composite control. After preparation, the specimens were allowed to mature for 24 hours prior to submitting to chewing simulation. The cumulative volumetric loss of restorative dental material against a standardized steatite antagonist was compared among the materials.

A null hypothesis was tested: Wear volumes of contemporary glass-ionomer containing materials advertised for use in posterior dentition as load-bearing posterior restorations have no difference compared to a contemporary resin composite. The null hypothesis for Equia Forte HT and Activa Bioactive Restorative was rejected. However, for Ionolux, the null hypothesis failed to reject.

At first glance, the advertisement of these materials appeal to the operative dentist given a combination of desirable qualities lending operator convenience and a potential for decreased technique sensitivity: An opportunity to bulk fill, optional adhesive or bonding protocols, and options to omit tooth substrate conditioning. The relatively large volume of glass-ionomer is amenable to service as a fluoride reservoir. Additionally, these materials are relatively esthetic in

comparison to amalgam and direct gold restorations. However, with limited available data to support clinical indications for use, the clinician may rely heavily on product advertisement.

Table 6. Summary of potential advantages of restorative materials used in this study.

	Surface conditioning required	Bonding protocol required	Can bulk fill (>2mm increments)
Activa Bioactive Restorative	Yes ★	Yes ★	Yes ★
Ionolux	No ★	No ★	No ★
Equia Forte HT	Yes ★	Yes ★	Yes ★

★ Clinical advantage ★ Clinical disadvantage

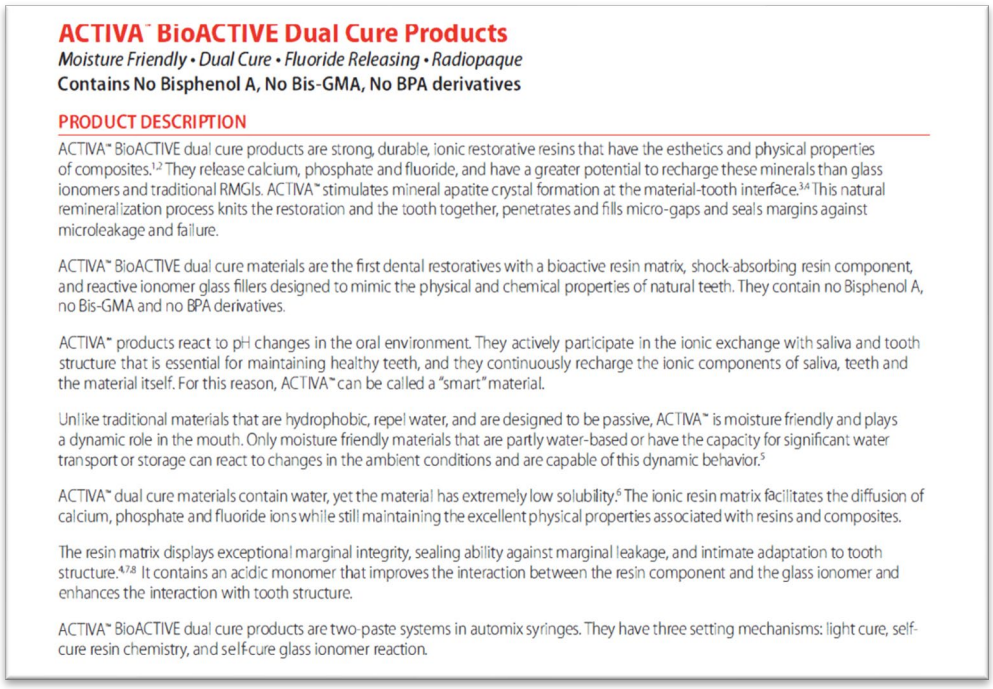
ACTIVA

Roulet et al. compared the wear of Activa Bioactive Restorative to a pure glass-ionomer, Fuji IX GP and found similar relationships between the material classes in the volumetric loss of material due to wear[134]. Under nearly identical conditions compared to this study, however, Roulet observed Activa to experience nearly three times the volumetric wear in mm^3. In another in vitro study, Latta et. al used an Alabama wear machine and subjected Activa, Equia Forte, Fuji II LC and an experimental self-adhesive restorative material to 400,000 cycles with a stainless steel ball antagonist using either photo-activated cure or self-cure mechanisms[135]. A similar trend observing a higher volumetric loss due to wear in Activa was identified when this material was permitted to self-cure only without photo-activation. A possible explanation of the observed difference is a lack of intentionally light-curing of the material, which is indicated according to manufacturer's instructions for use, and a stainless steel antagonist which is more aggressive

than steatite. Lacking proper photopolymerization where required may result in a decrease in mechanical properties[136] leading to increased observable wear.

To further investigate this idea, in a side experiment of this study, Activa Bioactive Restorative was generously dispensed using the manufacturer tips directly into a sealable plastic bag placed within a box crafted to prevent visible light entry in an attempt to investigate if the material is suitably a resin-modified glass-ionomer. The material was permitted to self-cure undisturbed for 72 hours in the dark. After time had elapsed, the material was evaluated for surface consistency while still inside the bag and light-sealed box. The material was of a relatively soft mass and still compressible and fluid between the fingers, revealing presumably major uncured portions still within the mixture. The bag containing the material was then removed from the box for direct observation. The uncured portion was whiter in color and retained consistency close to flowable resin composite, while a relatively harder mass was pale yellow. The sample was then light-cured through the plastic bag and re-evaluated for surface consistency. The restorative material was now hardened throughout the entirety of the mass.

Fig 20. Pulpdent product description for Activa Bioactive Restorative.



ACTIVA™ BioACTIVE Dual Cure Products

Moisture Friendly • Dual Cure • Fluoride Releasing • Radiopaque

Contains No Bisphenol A, No Bis-GMA, No BPA derivatives

PRODUCT DESCRIPTION

ACTIVA™ BioACTIVE dual cure products are strong, durable, ionic restorative resins that have the esthetics and physical properties of composites.[1,2] They release calcium, phosphate and fluoride, and have a greater potential to recharge these minerals than glass ionomers and traditional RMGIs. ACTIVA™ stimulates mineral apatite crystal formation at the material-tooth interface.[3,4] This natural remineralization process knits the restoration and the tooth together, penetrates and fills micro-gaps and seals margins against microleakage and failure.

ACTIVA™ BioACTIVE dual cure materials are the first dental restoratives with a bioactive resin matrix, shock-absorbing resin component, and reactive ionomer glass fillers designed to mimic the physical and chemical properties of natural teeth. They contain no Bisphenol A, no Bis-GMA and no BPA derivatives.

ACTIVA™ products react to pH changes in the oral environment. They actively participate in the ionic exchange with saliva and tooth structure that is essential for maintaining healthy teeth, and they continuously recharge the ionic components of saliva, teeth and the material itself. For this reason, ACTIVA™ can be called a "smart" material.

Unlike traditional materials that are hydrophobic, repel water, and are designed to be passive, ACTIVA™ is moisture friendly and plays a dynamic role in the mouth. Only moisture friendly materials that are partly water-based or have the capacity for significant water transport or storage can react to changes in the ambient conditions and are capable of this dynamic behavior.[5]

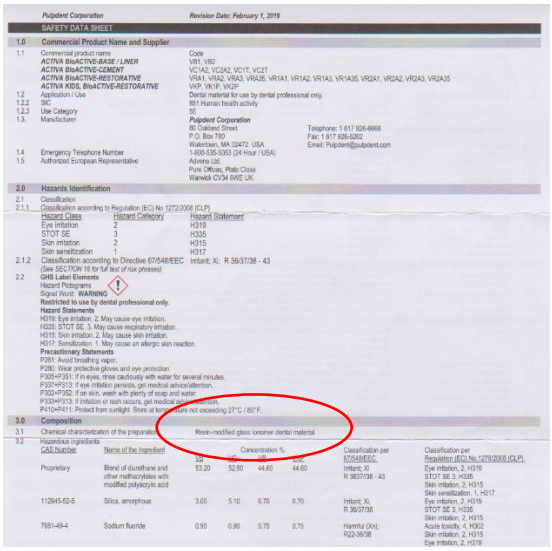
ACTIVA™ dual cure materials contain water, yet the material has extremely low solubility.[6] The ionic resin matrix facilitates the diffusion of calcium, phosphate and fluoride ions while still maintaining the excellent physical properties associated with resins and composites.

The resin matrix displays exceptional marginal integrity, sealing ability against marginal leakage, and intimate adaptation to tooth structure.[4,7,8] It contains an acidic monomer that improves the interaction between the resin component and the glass ionomer and enhances the interaction with tooth structure.

ACTIVA™ BioACTIVE dual cure products are two-paste systems in automix syringes. They have three setting mechanisms: light cure, self-cure resin chemistry, and self-cure glass ionomer reaction.

The manufacturer claims regarding Activa Bioactive Restorative are remarkable. Of interest to this study is the composition and setting chemistry. The product description explicitly states a triple setting mechanism, including self-cure glass-ionomer reaction in addition to self-cure resin and light cure resin. Additionally, based on the industry-required safety data sheets, the manufacturer has changed the product description from "resin-modified glass ionomer dental material" to "bioactive ionic resin with reactive glass filler." Some authors suggest that this material could be considered a resin-modified glass-ionomer but not a resin composite[137]. Although there was seemingly no difference other than product description on the safety data sheets between 2019 and 2020, these findings along with the results of the side experiment in this study would indicate that Activa Bioactive Restorative is not a resin-modified glass-ionomer according to traditional nomenclature based on an inability of the material to fully cure in the absence of light-activation.

Fig 21. Activa Safety Data Sheets dated February 2019 (left) and July 2019 (right). Block three includes manufacturer-provided chemical characteristic of the product.



Pulpdent Corporation — Revision Date: February 1, 2019

SAFETY DATA SHEET

1.0 Commercial Product Name and Supplier

1.1	Commercial product name	Code
	ACTIVA BioACTIVE-BASE / LINER	VB1, VB2
	ACTIVA BioACTIVE-CEMENT	VC1A2, VC2A2, VC1T, VC2T
	ACTIVA BioACTIVE-RESTORATIVE	VRA1, VRA2, VRA3, VRA35, VR1A1, VR1A2, VR1A3, VR1A35, VR2A1, VR2A2, VR2A3, VR2A35
	ACTIVA KIDS, BioACTIVE-RESTORATIVE	VKP, VK1P, VK2P
1.2	Application / Use	Dental material for use by dental professional only.
1.2.2	SIC	851 Human health activity
1.2.3	Use Category	55
1.3.	Manufacturer	Pulpdent Corporation 80 Oakland Street P.O. Box 780 Watertown, MA 02472 USA Telephone: 1 617 926-6666 Fax: 1 617 926-0262 Email: Pulpdent@pulpdent.com
1.4	Emergency Telephone Number	1-800-535-5053 (24 hour / USA)
1.5	Authorized European Representative	Advena Ltd. Pure Offices, Plato Close Warwick CV34 6WE UK

2.0 Hazards Identification

2.1 Classification

2.1.1 Classification according to Regulation (EC) No 1272/2008 [CLP]

Hazard Class	Hazard Category	Hazard Statement
Eye irritation	2	H319
STOT SE	3	H335
Skin irritation	2	H315
Skin sensitization	1	H317

2.1.2 Classification according to Directive 67/548/EEC — Irritant; Xi; R 36/37/38 - 43

(See SECTION 16 for full text of risk phrases)

2.2 GHS Label Elements

Hazard Pictograms

Signal Word: WARNING

Restricted to use by dental professional only.

Hazard Statements

H319: Eye irritation. 2. May cause eye irritation.
H335: STOT SE. 3. May cause respiratory irritation.
H315: Skin irritation. 2. May cause skin irritation.
H317: Sensitization. 1. May cause an allergic skin reaction.

Precautionary Statements

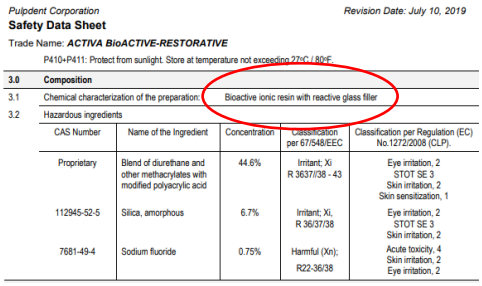
P261: Avoid breathing vapor.
P280: Wear protective gloves and eye protection.
P305+P351: If in eyes, rinse cautiously with water for several minutes.
P337+P313: If eye irritation persists, get medical advice/attention.
P302+P352: If on skin, wash with plenty of soap and water.
P333+P313: If irritation or rash occurs, get medical advice/attention.
P410+P411: Protect from sunlight. Store at temperatures not exceeding 27°C / 80°F.

3.0 Composition

3.1 Chemical characterization of the preparation: Resin-modified glass ionomer dental material

3.2 Hazardous ingredients

CAS Number	Name of the Ingredient	Concentration %				Classification per 67/548/EEC	Classification per Regulation (EC) No.1272/2008 (CLP)
		VB	VC	VR	VK		
Proprietary	Blend of diurethane and other methacrylates with modified polyacrylic acid	53.20	52.90	44.60	44.60	Irritant; Xi R 36/37/38 - 43	Eye irritation, 2. H319 STOT SE 3. H335 Skin irritation, 2. H315 Skin sensitization, 1. H317
112945-52-5	Silica, amorphous	3.00	5.10	6.70	6.70	Irritant; Xi, R 36/37/38	Eye irritation, 2. H319 STOT SE 3. H335 Skin irritation, 2. H315
7681-49-4	Sodium fluoride	0.90	0.90	0.75	0.75	Harmful (Xn); R22-36/38	Acute toxicity, 4. H302 Skin irritation, 2. H315 Eye irritation, 2. H319



Pulpdent Corporation — Revision Date: July 10, 2019

Safety Data Sheet

Trade Name: ***ACTIVA BioACTIVE-RESTORATIVE***

P410+P411: Protect from sunlight. Store at temperature not exceeding 27°C / 80°F.

3.0 Composition

3.1 Chemical characterization of the preparation: Bioactive ionic resin with reactive glass filler

3.2 Hazardous ingredients

CAS Number	Name of the Ingredient	Concentration	Classification per 67/548/EEC	Classification per Regulation (EC) No.1272/2008 (CLP).
Proprietary	Blend of diurethane and other methacrylates with modified polyacrylic acid	44.6%	Irritant; Xi R 3637/38 - 43	Eye irritation, 2 STOT SE 3 Skin irritation, 2 Skin sensitization, 1
112945-52-5	Silica, amorphous	6.7%	Irritant; Xi, R 36/37/38	Eye irritation, 2 STOT SE 3 Skin irritation, 2
7681-49-4	Sodium fluoride	0.75%	Harmful (Xn); R22-36/38	Acute toxicity, 4 Skin irritation, 2 Eye irritation, 2

Bansal et. al compared Activa Bioactive Restorative to Filtek Supreme Ultra, Fuji IX and Fuji II but under slightly different in vitro conditions; only a 20N force, 100,000 testing cycles and no thermocycling were performed to reveal no statistically significant difference between Activa and Filtek Supreme Ultra, but both experienced statistically significantly lower wear than Fuji IX and Fuji II[138]. These findings are partially supported by this study, in that glass-ionomer and resin-modified glass-ionomers experience increased relative wear but that Activa Bioactive Restorative wore less than the resin composite Filtek Supreme Ultra. Also, the amount of wear is comparable, most likely the result of photopolymerization of the samples. The relative amount of wear in Activa is reasonably explained with consideration to the differences in parameters.

EQUIA FORTE HT

Equia Forte HT experienced the largest mean volumetric loss of material due to wear at all chewing cycle intervals. The manufacturer-provided imaging suggests the resin coating is no more than 10 micrometers thick (FIG X—below). Based on the SEM images made of Equia Forte HT samples in this study, this resin coating was obliterated under load of the antagonist. The manufacturer does acknowledge an expectation that the resin coating will wear away in approximately six months to one year, and during this time the glass-ionomer polygel matrix will undergo maturation to further increase mechanical properties. Although matrix maturation and resultant increase in mechanical properties can be expected over time[139], it is more likely based on the findings from this study that the coating would be lost much earlier when comparing the wear facet to the approximately 10 micrometer-thick resin coating (Fig X below).

Fig 22. (a) Product description and manufacturer provided SEM of Equia Forte HT and Coat. (b) SEM of Equia Forte wear facet profile in this study. Note ten times difference in scale of magnification.

EQUIA Forte HT is a glass hybrid restorative system that combines a self-cure bulk fill restorative (EQUIA Forte HT Fil) with a highly filled, light-cure resin coating agent (EQUIA Forte Coat) (Fig. 1).

(a)

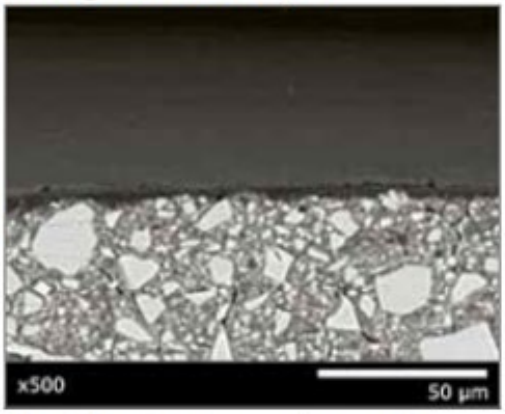
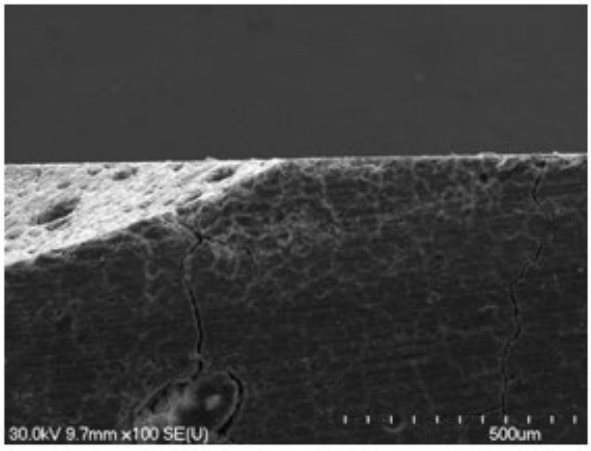


Figure 1: Scanning electron microscopy of EQUIA Forte HT, comprising of EQUIA Forte HT Fil covered with EQUIA Forte Coat.
Source: GC R&D, Japan, 2020

(b)



IONOLUX

A vast majority of data concerning the in vitro volumetric wear of resin-modified glass-ionomers suggest the material as a category performs in between resin composites and pure glass-ionomers with regards to linear and volumetric wear[140]. The findings of this study are

inconsistent in this regard, in that the resin-modified glass-ionomer performed indistinguishably from the resin composite control over time. A possible rationale is such that the proprietary formulation lends the material increased resistance to wear. However, the exact chemical composition is secret at this time. Regarding an increased early rate of wear compare to resin composite control, a possible explanation is the phenomenon called the "running in" period, in which very early intervals might be unreliable[141]. Perhaps the more significant relationship is the relative wear amongst the four groups that remains relatively consistent over time. At all times, the resin-modified glass-ionomer Ionolux outperformed the glass-ionomer hybrid system Equia Forte HT.

As the Ionolux material does not reportedly require a tooth substrate conditioning procedure or bonding protocol, the only seeming limitation may be the inability to bulk fill in the posterior region. The material should only be placed in increments not to exceed two millimeters according to the manufacturer. This might lend the material to decrease technique sensitivity and could be advantageous. Pediatric settings, for example, may benefit from this material.

FILTEK SUPREME ULTRA

Correlation to clinical performance of in vitro studies remains a top priority for translating findings from the laboratory to the clinic. When the Filtek Supreme product line was launched by 3M in the early 2000's, there were still clinical trials measuring in vivo wear as a focus of concern on this topic. Although not exactly the same product as tested in this study, Palaniappan et al. observed similar total surface volume loss ($0.60mm^3$) after two years of clinical service[142] which was comparable to the estimated service time and volumetric wear of this in vitro study ($0.78mm^3$). With reasonable consideration, these absolute values are not

magnitudes apart, permitting some credence to the external validity of this study and the chewing simulator parameters.

8. Limitations of the study

This study focused on wear comparison amongst glass ionomer containing restorative materials and a resin composite over a simulated two years of clinical service in a chewing simulator. For convenience and consistency, steatite was used in lieu of a natural human tooth antagonist. Although an enamel substrate might be more clinically relevant, a commercially available steatite antagonist is more advantageous based on reduced variability and replicability. The specimens lacked anatomic form in order to produce wear on a standardized surface and subsequent feasibility to measure resulting wear volumes. In vitro chewing simulators are subject to variability and known to be difficult to correlate to other in vitro studies and clinical performance[143].

Although the gold-standard for clinical research remains randomized clinical trials, long-term in vivo studies may largely be impractical for every new restorative dental material. The typical challenges associated with these undertaking, including cost, patient drop-out, ethical issues, and longevity are largely eclipsed by the incredible rate at which new materials are made available. The ability to rapidly identify differences in wear resistance lend credence to chewing simulators as a useful tool to identify new restorative materials for merit and further, more expensive, clinical investigations. A more practical approach to dental materials evaluation would involve the use of correlated chewing simulations in order to rapidly identify products with merit for further investigation. Some authors believe the multiple, ongoing enhancements of glass ionomer containing restorative materials may contend with conventional restorative

materials such as amalgam and resin composite[144]. The evidence in this study reveals this potential may exist, but further investigation is warranted.

9. Conclusion

Measurable differences in volumetric wear are found when comparing materials advertised for use as posterior load-bearing restorations within and across material classes and combinations of restorative dental materials when fatigued in a chewing simulator.

Even though American Dental Association specifications for wear of posterior load-bearing restorations have been retired, clinical significance of the observed in vitro differences of the glass-ionomer containing materials investigated in this study should be anticipated. The glass-ionomer hybrid system wears two times faster than a well-accepted resin composite.

Resin-coatings of glass-ionomers are difficult to control in thickness and are likely impractical, as they would be swiftly worn away before matrix maturation can occur resulting in likely unacceptable wear of the underlying glass-ionomer.

Dental manufacturers exercise liberties in product advertisement. Current nomenclature to describe dental restorative materials is equally liberal, nondescript or potentially misleading. Dentists should approach various contemporary formulations advertised as “novel” with caution. Activa Bioactive Restorative is not a resin-modified glass-ionomer. With a product requirement to use a dentin-bonding agent negating a potential for bioactive interactions with the tooth substrate, the decreased wear rates of Activa Bioactive Restorative may not be a clinical advantage compared to resin composite alternatives.

The resin-modified glass-ionomer Ionolux should be evaluated in further chewing simulations and mechanical properties investigated prior to consideration in clinical trials to determine suitability as a definitive posterior load-bearing restorative material.

REFERENCES

1. Wilson AD. A new translucent cement for dentistry: the glass-ionomer cement. Br Dent J. 1972;132:133-5.

2. Wilson AD, McLean JW. Clinical uses. Glass Ionomer Cements. Chicago, Quintessence Publication. 1988.

3. ISO 9917–1: Dental Water Based Cements; International Organization for Standardization: Geneva, Switzerland, 2003.

4. Albers HF. Tooth-colored restoratives: principles and techniques. PMPH-USA; 2002.

5. Mclean JW. Proposed nomenclature for glass-ionomer dental cements and related materials. Quintessence Int. 1994;25:587-9.

6. Black GV. A Work on Operative Dentistry in Two Volumes. Medico-dental publishing Company; 1920.

7. Stefanac SJ, Nesbit SP. Treatment Planning in Dentistry-E-Book. Elsevier Health Sciences; 2006 Aug 29.

8. Roberson T, Heymann HO, Swift Jr EJ. Sturdevant's art and science of operative dentistry. Elsevier Health Sciences; 2006 Apr 13.

9. Stanley HR, Going RE, Chauncey HH. Human pulp response to acid pretreatment of dentin and to composite restoration. The Journal of the American Dental Association. 1975 Oct 1;91(4):817-25.

10. Cox CF, Keall CL, Keall HJ, Ostro E, Bergenholtz G. Biocompatibility of surface-sealed dental materials against exposed pulps. Journal of Prosthetic Dentistry. 1987 Jan 1;57(1):1-8.

11. Hilton TJ. Keys to clinical success with pulp capping: a review of the literature. Operative dentistry. 2009 Sep;34(5):615-25.

12. Van Dijken JW, Pallesen U. Long-term dentin retention of etch-and-rinse and self-etch adhesives and a resin-modified glass ionomer cement in non-carious cervical lesions. Dental Materials. 2008 Jul 1;24(7):915-22.

13. Tyas MJ. Clinical evaluation of a resin-modified glass-ionomer adhesive system, results at five years Operative Dent 2002, 27:438-41.

14. Koubi S, Raskin A, Bukiet F, Pignoly C, Toca E, Tassery H. One-year clinical evaluation of two resin composites, two polymerization methods, and a resin-modified glass ionomer in non-carious cervical lesions. J Contemp Dent Pract. 2006 Nov 1;7(5):42-53.

15. Peumans M, Kanumilli P, Munck De J, Landuyt Van K, Lambrechts P, Meerbeek Van B. Clinical effectiveness of contemporary adhesives: A systematic review of current clinical trials. Journal of Esthetic and Restorative Dentistry. 2010;22(1):73-4.

16. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic?. Journal-Canadian Dental Association. 2003 Dec;69(11):726-32.

17. KJ A. Phillips Science of dental materials 11th ed. St. Louis: Saunders. 2003:89-90.

18. McLean JW, Wilson AD. The clinical development of the glass-ionomer cements. I. Formulations and properties. Australian Dental Journal. 1977 Feb;22(1):31-6.

19. Sawani S, Arora V, Jaiswal S, Nikhil V. Comparative evaluation of microleakage in Class II restorations using open vs. closed centripetal build-up techniques with different lining materials. Journal of conservative dentistry: JCD. 2014 Jul;17(4):344.

20. Fabianelli A, Sgarr A, Goracci C, Cantoro A, Pollington S, Ferrari M. Microleakage in class II restorations: open vs closed centripetal build-up technique. Operative Dentistry. 2010 May;35(3):308-13.

21. Kydd WL, Nicholls JI, Harrington G, Freeman M. Marginal leakage of cast gold crowns luted with zinc phosphate cement: an in vivo study. The Journal of prosthetic dentistry. 1996 Jan 1;75(1):9-13.

22. McLean JM. The use of glass-ionomer cements in bonding composite resins to dentine. Br Dent J. 1985;158:410-4.

23. Frankenberger R, Garcia-Godoy F, Krämer N. Clinical performance of viscous glass ionomer cement in posterior cavities over two years. International Journal of Dentistry. 2009 Jan 1;2009.

24. Basso M, Brambilla E, Benites M, Giovannardi M, Ionescu A. Glassionomer cement for permanent dental restorations: a 48-months, multi-centre, prospective clinical trial.

25. Klinke T, Daboul A, Turek A, Frankenberger R, Hickel R, Biffar R. Clinical performance during 48 months of two current glass ionomer restorative systems with coatings: a randomized clinical trial in the field. Trials. 2016 Dec 1;17(1):239.

26. van Dijken JW, Pallesen U, Benetti A. A randomized controlled evaluation of posterior resin restorations of an altered resin modified glass-ionomer cement with claimed bioactivity. Dental Materials. 2019 Feb 1;35(2):335-43.

27. Gurgan S, Kutuk ZB, Ozturk C, Soleimani R, Cakir FY. Clinical Performance of a Glass Hybrid Restorative in Extended Size Class II Cavities. Operative Dentistry. 2019 Oct 29.

28. Balkaya H, Arslan S. A Two-year Clinical Comparison of Three Different Restorative Materials in Class II Cavities. Operative Dentistry. 2020 Jan;45(1):E32-42.

29. Gurgan S, Kutuk ZB, Cakir FY, Ergin E. A randomized controlled 10 years follow up of a glass ionomer restorative material in class I and class II cavities. Journal of dentistry. 2020 Mar 1;94:103175.

30. Frencken JE, Pilot T, Songpaisan Y, Phantumvanit P. Atraumatic restorative treatment (ART): rationale, technique, and development. Journal of public health dentistry. 1996 Sep;56(3):135-40.

31. Barnes DM, Blank LW, Gingell JC, Gilnder PP. A clinical evaluation of a resin-modified: glass ionomer restorative material. The Journal of the American Dental Association. 1995 Sep 1;126(9):1245-53.

32. Mobarak E, El-Deeb H, Daifalla LE, Ghaly M, Mustafa M, Sabry D, Fatah H, Mulder J, Creugers NH, Frencken JE. Survival of multiple-surface ART restorations using a zinc-reinforced glass-ionomer restorative after 2 years: A randomized triple-blind clinical trial. Dental Materials. 2019 Sep 1;35(9):e185-92.

33. Frencken JE, Leal SC, Navarro MF. Twenty-five-year atraumatic restorative treatment (ART) approach: a comprehensive overview. Clinical oral investigations. 2012 Oct 1;16(5):1337-46.

34. Dorri M, Martinez-Zapata MJ, Walsh T, Marinho VC, Sheiham A, Zaror C. Atraumatic restorative treatment versus conventional restorative treatment for managing dental caries. Cochrane Database of Systematic Reviews. 2017(12).

35. Toh SL, Messer LB. Evidence-based assessment of tooth-colored restorations in proximal lesions of primary molars. Pediatric Dentistry. 2007 Jan 1;29(1):8-15.

36. Chadwick BL, Evans DJ. Restoration of class II cavities in primary molar teeth with conventional and resin modified glass ionomer cements: a systematic review of the literature. European Archives of Paediatric Dentistry. 2007 Mar 1;8(1):14-21.

37. Dermata A, Papageorgiou SN, Fragkou S, Kotsanos N. Comparison of resin modified glass ionomer cement and composite resin in class II primary molar restorations: a 2-year parallel randomised clinical trial. European Archives of Paediatric Dentistry. 2018 Dec 1;19(6):393-401.

38. Ma X, Li C, Jia L, Wang Y, Liu W, Zhou X, Johnson TM, Huang D. Materials for retrograde filling in root canal therapy. Cochrane Database of Systematic Reviews. 2016(12).

39. Beauchamp J, Caufield PW, Crall JJ, Donly K, Feigal R, Gooch B, Ismail A, Kohn W, Siegal M, Simonsen R. Evidence-based clinical recommendations for the use of pit-and-fissure sealants: a report of the American Dental Association Council on Scientific Affairs. The Journal of the American Dental Association. 2008 Mar 1;139(3):257-68.

40. Simonsen RJ. Chapter 2: Pit and fissure sealants. Clinical Applications of the Acid Etch Technique. 1st ed. Chicago, IL: Quintessence Publishing Co, Inc. 1978:19-42.

41. Ahovuo-Saloranta A, Forss H, Walsh T, Nordblad A, Mäkelä M, Worthington HV. Pit and fissure sealants for preventing dental decay in permanent teeth. Cochrane database of systematic reviews. 2017(7).

42. Eames WB, O'Neal SJ, Monteiro J, Miller C, Roan JD, Cohen KS. Techniques to improve the seating of castings. The Journal of the American Dental Association. 1978 Mar 1;96(3):432-7.

43. Wilson Jr TG. The positive relationship between excess cement and peri-implant disease: a prospective clinical endoscopic study. Journal of periodontology. 2009 Sep;80(9):1388-92.

44. Wadhwani C, Hess T, Faber T, Piñeyro A, Chen CS. A descriptive study of the radiographic density of implant restorative cements. The Journal of prosthetic dentistry. 2010 May 1;103(5):295-302.

45. Millett DT, Glenny AM, Mattick RC, Hickman J, Mandall NA. Adhesives for fixed orthodontic bands. Cochrane Database of Systematic Reviews. 2016(10).

46. Wilson AD. A hard decade's work: steps in the invention of the glass-ionomer cement. Journal of dental research. 1996 Oct;75(10):1723-7.

47. McClean 1988 Glass-ionomer cements. British Dental Journal 164:293.

48. McCabe JF, JF M, PA J. 1978 Some properties of a glass ionomer cement.

49. Charlton DG, Moore BK, Swartz ML. Direct surface pH determinations of setting cements. Operative Dentistry. 1991 Nov 1;16(6):231-8.

50. Saito, S Tosaki S Hirota K Advances in glass ionomer cements quintessence publishing co: berlin Germany 1999 p15-50.

51. Wilson and McLean 1988. Glass-ionomer cement. Chicago: quintessence publishing.

52. Nicholson JW. Maturation processes in glass-ionomer dental cements. Acta biomaterialia odontologica Scandinavica. 2018 Jan 1;4(1):63-71.

53. Nicholson JW, Croll TP. Glass-ionomer cements in restorative dentistry. Quintessence international. 1997 Nov 1;28(11).

54. Najeeb S, Khurshid Z, Zafar MS, Khan AS, Zohaib S, Martí JM, Sauro S, Matinlinna JP, Rehman IU. Modifications in glass ionomer cements: nano-sized fillers and bioactive nanoceramics. International journal of molecular sciences. 2016 Jul;17(7):1134.

55. Khoroushi M, Keshani F. A review of glass-ionomers: From conventional glass-ionomer to bioactive glass-ionomer. Dental research journal. 2013 Jul;10(4):411.

56. Croll TP, Berg JH, Donly KJ. Dental repair material: a resin-modified glass-ionomer bioactive ionic resin-based composite. Compend Contin Educ Dent. 2015 Jan 1;36(1):60-5.

57. Schwendicke F, Göstemeyer G, Blunck U, Paris S, Hsu LY, Tu YK. Directly placed restorative materials: review and network meta-analysis. Journal of Dental Research. 2016 Jun;95(6):613-22.

58. Wu CH, Smales RJ. Occlusal wear of a resin-modified glass ionomer cement following three surface treatments. Clinical oral investigations. 2001 Mar 1;5(1):26-30.

59. Kato K, Yarimizu H, Nakaseko H, Sakuma T. Influence of coating materials on conventional glass-ionomer cement. J. Dent. Res. 2008;87(487):1996-44.

60. Fuhrmann D, Murchison D, Whipple S, Vandewalle K. Properties of New Glass-Ionomer Restorative Systems Marketed for Stress-Bearing Areas. Operative Dentistry. 2020 Jan;45(1):104-10.

61. Crisp S, Lewis BG, Wilson AD. Characterization of glass-ionomer cements: 2. Effect of the powder: liquid ratio on the physical properties. Journal of Dentistry. 1976 Nov 1;4(6):287-90.

62. Crisp S, Wilson AD. Reactions in glass ionomer cements: V. Effect of incorporating tartaric acid in the cement liquid. Journal of Dental Research. 1976 Nov;55(6):1023-31.

63. Sulaiman TA, Abdulmajeed AA, Altitinchi A, Ahmed SN, Donovan TE. Physical Properties, Film Thickness, and Bond Strengths of Resin-Modified Glass Ionomer Cements According to Their Delivery Method. Journal of Prosthodontics. 2019 Jan;28(1):85-90.

64. Nicholson JW. Maturation processes in glass-ionomer dental cements. Acta biomaterialia odontologica Scandinavica. 2018 Jan 1;4(1):63-71.

65. Moberg M, Brewster J, Nicholson J, Roberts H. Physical property investigation of contemporary glass ionomer and resin-modified glass ionomer restorative materials. Clinical Oral Investigations. 2019 Mar 8;23(3):1295-308.

66. Baier RE. Principles of adhesion. Operative dentistry. 1992 Jan 1:1-9.

67. Bowen RL. Use of epoxy resins in restorative materials. Journal of Dental Research. 1956 Jun;35(3):360-9.

68. Wilder Jr AD, Swift Jr EJ, Heymann HO, Ritter AV, Sturdevant JR, Bayne SC. A 12-year clinical evaluation of a three-step dentin adhesive in noncarious cervical lesions. The Journal of the American Dental Association. 2009 May 1;140(5):526-35.

69. Chee B, Rickman LJ, Satterthwaite JD. Adhesives for the restoration of non-carious cervical lesions: a systematic review. Journal of Dentistry. 2012 Jun 1;40(6):443-52.

70. Peumans M, De Munck J, Mine A, Van Meerbeek B. Clinical effectiveness of contemporary adhesives for the restoration of non-carious cervical lesions. A systematic review. Dental Materials. 2014 Oct 1;30(10):1089-103.

71. Nicholson JW. Adhesion of glass-ionomer cements to teeth: a review. International Journal of Adhesion and Adhesives. 2016 Sep 1;69:33-8.

72. Wilson AD, Prosser HJ, Powis DM. Mechanism of adhesion of polyelectrolyte cements to hydroxyapatite. Journal of dental research. 1983 May;62(5):590-2.

73. Degrange M, Roulet JF, editors. Minimally invasive restorations with bonding. Quintessence Publishing (IL); 1997.

74. Sidhu SK. Glass-ionomer cement restorative materials: a sticky subject?. Australian dental journal. 2011 Jun;56:23-30.

75. Lin A, McIntyre NS, Davidson RD. Studies on the adhesion of glass-ionomer cements to dentin. Journal of dental research. 1992 Nov;71(11):1836-41.

76. Inoue S, Van Meerbeek B, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, Sano H. Effect of remaining dentin thickness and the use of conditioner on micro-tensile bond strength of a glass-ionomer adhesive. Dental Materials. 2001 Sep 1;17(5):445-55.

77. Gilboe DB, Svare CW, Thayer KE, Drennon DG. Dentinal smearing: an investigation of the phenomenon. Journal of Prosthetic Dentistry. 1980 Sep 1;44(3):310-6.

78. Tay FR, Smales RJ, Ngo H, Wei SH, Pashley DH. Effect of different conditioning protocols on adhesion of a GIC to dentin. Journal of Adhesive Dentistry. 2001 Jun 1;3(2).

79. Powis DR, Folleråas T, Merson SA, Wilson AD. Materials science: Improved adhesion of a glass ionomer cement to dentin and enamel. Journal of Dental Research. 1982 Dec;61(12):1416-22.

80. Barakat MM, Powers JM, Yamaguchi R. Parameters that affect in vitro bonding of glass-ionomer liners to dentin. Journal of dental research. 1988 Sep;67(9):1161-3.

81. Armstrong SR, Boyer DB, Keller JC. Microtensile bond strength testing and failure analysis of two dentin adhesives. Dental Materials. 1998 Jan 1;14(1):44-50.

82. Yip HK, Tay FR, Ngo HC, Smales RJ, Pashley DH. Bonding of contemporary glass ionomer cements to dentin. Dental Materials. 2001 Sep 1;17(5):456-70.

83. Mclean JW. A new method of bonding dental cements and porcelain to metal surfaces. Oper Dent. 1977;2:130-42.

84. Hinoura K, Moore BK, Phillips RW. Tensile bond strength between glass ionomer cements and composite resins. The Journal of the American Dental Association. 1987 Feb 1;114(2):167-72.

85. Pameijer CH, Garcia-Godoy F, Morrow BR, Jefferies SR. Flexural strength and flexural fatigue properties of resin-modified glass ionomers. J Clin Dent. 2015 Jan 1;26(1):23-7.

86. Standard I. ISO 4049 polymer based filling, restorative and luting materials.

87. International Organization for Standardization. 2000;1:27.

88. Ferracane JL. Correlating abrasive wear to mechanical properties of experimental dental composites. InTransactions of third international congress on dental materials 1977 (Vol. 319).

89. Xu X, Burgess JO. Compressive strength, fluoride release and recharge of fluoride-releasing materials. Biomaterials. 2003 Jun 1;24(14):2451-61.

90. Anusavice KJ, Shen C, Rawls HR, editors. Phillips' science of dental materials. Elsevier Health Sciences; 2012 Sep 27.

91. Cenci MS, Pereira-Cenci T, Donassollo TA, Sommer L, Strapasson A, Demarco FF. Influence of thermal stress on marginal integrity of restorative materials. Journal of Applied Oral Science. 2008 Apr;16(2):106-10.

92. Swift Jr EJ. Fluoride release from two composite resins. Quintessence international (Berlin, Germany: 1985). 1989 Dec;20(12):895.

93. Geiger SB, Weiner S. Fluoridated carbonatoapatite in the intermediate layer between glass ionomer and dentin. Dental Materials. 1993 Jan 1;9(1):33-6.

94. Olsen BT, Garcia-Godoy F, Marshall TD, Barnwell GM. Fluoride release from glass ionomer-lined amalgam restorations. Am J Dent. 1989 Jun;2(3):89-91.

95. Cildir SK, Sandalli N. Fluoride release/uptake of glass-ionomer cements and polyacid-modified composite resins. Dental materials journal. 2005;24(1):92-7.

96. Peng D, Smales RJ, Yip HK, Shu M. In vitro fluoride release from aesthetic resorative materials following recharging with APF gel. Australian dental journal. 2000 Sep;45(3):198-203.

97. Earl MS, Hume WR, Mount GJ. Effect of varnishes and other surface treatments on water movement across the glass-ionomer cement surface. Australian dental journal. 1985 Aug;30(4):298-301.

98. Brzović Rajić V, Miletić I, Gurgan S, Peroš K, Verzak Ž, Ivanišević Malčić A. Fluoride release from glass ionomer with nano filled coat and varnish. Acta stomatologica Croatica. 2018 Dec 19;52(4):307-13.

99. Della Bona A, Watts DC. Evidence-based dentistry and the need for clinically relevant models to predict material performance. Dental materials: official publication of the Academy of Dental Materials. 2013 Jan;29(1):1.

100. Lyons MF, Cadden SW, Baxendale RH, Yemm R. Twitch interpolation in the assessment of the maximum force-generating capacity of the jaw-closing muscles in man. Archives of oral biology. 1996 Dec 1;41(12):1161-8.

101. Gibbs CH, Mahan PE, Lundeen HC, Brehnan K, Walsh EK, Holbrook WB. Occlusal forces during chewing and swallowing as measured by sound transmission. Journal of Prosthetic Dentistry. 1981 Oct 1;46(4):443-9.

102. Gibbs CH, Anusavice KJ, Young HM, Jones JS, Esquivel-Upshaw JF. Maximum clenching force of patients with moderate loss of posterior tooth support: a pilot study. The Journal of prosthetic dentistry. 2002 Nov 1;88(5):498-502.

103. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. Journal of dentistry. 1999 Feb 1;27(2):89-99.

104. Ferracane JL. Is the wear of dental composites still a clinical concern?: Is there still a need for in vitro wear simulating devices?. Dental Materials. 2006 Aug 1;22(8):689-92.

105. Swift EJ. Wear of composite resins in permanent posterior teeth. The Journal of the American Dental Association. 1987 Oct 1;115(4):584-8.

106. Lambrechts P, Braem M, Vuylsteke-Wauters M, Vanherle G. Quantitative in vivo wear of human enamel. Journal of dental research. 1989 Dec;68(12):1752-4.

107. Journal of the American Dental Association. 2003;134:510.

108. Anusavice KJ, Shen C, Rawls HR, editors. Phillips' science of dental materials. Elsevier Health Sciences; 2012.

109. Kunzelmann KH. Glass-ionomer cements, cermet cements,“hybrid”-glass-ionomers and compomers–laboratory trials–wear resistance. Trans. Acad. Dent. Mater. 1996;9:89-104.

110. De Gee AJ, Van Duinen RN, Werner A, Davidson CL. Early and long-term wear of conventional and resin-modified glass ionomers. Journal of dental research. 1996 Aug;75(8):1613-9.

111. American Dental Association. New American Dental Association: Specification No. 27 for Polymer Based Restorative Materials. American Dental Association; 2016.

112. Mair LH, Stolarski TA, Vowles RW, Lloyd CH. Wear: mechanisms, manifestations and measurement. Report of a workshop. Journal of dentistry. 1996 Jan 1;24(1-2):141-8.

113. Han JM, Zhang H, Choe HS, Lin H, Zheng G, Hong G. Abrasive wear and surface roughness of contemporary dental composite resin. Dental materials journal. 2014 Jul 30:2013-339.

114. Tsujimoto A, Barkmeier WW, Fischer NG, Nojiri K, Nagura Y, Takamizawa T, Latta MA, Miazaki M. Wear of resin composites: Current insights into underlying mechanisms, evaluation methods and influential factors. Japanese Dental Science Review. 2018 May 1;54(2):76-87.

115. Oh WS, DeLong R, Anusavice KJ. Factors affecting enamel and ceramic wear: a literature review. The Journal of prosthetic dentistry. 2002 Apr 1;87(4):451-9.

116. American Dental Association website www.ada.org./en/member-center/oral-health-topics/toothpastes 2020.

117. Zhang Y, Sailer I, Lawn BR. Fatigue of dental ceramics. Journal of dentistry. 2013 Dec 1;41(12):1135-47.

118. Lagouvardos P, Nikolinakos N, Oulis C. Volume fraction and location of voids and gaps in ultraconservative restorations by X-ray computed micro-tomography. Dental research journal. 2015 Nov;12(6):520.

119. Bartlett D, Ganss C, Lussi A. Basic Erosive Wear Examination (BEWE): a new scoring system for scientific and clinical needs. Clinical oral investigations. 2008 Mar 1;12(1):65-8.

120. Eccles JD, Jenkins WG. Dental erosion and diet. Journal of Dentistry. 1974 Jul 1;2(4):153-9.

121. Young WG, Khan F. Sites of dental erosion are saliva-dependent. Journal of oral rehabilitation. 2002 Jan;29(1):35-43.

122. Lambrechts P, Goovaerts K, Bharadwaj D, De Munck J, Bergmans L, Peumans M, Van Meerbeek B. Degradation of tooth structure and restorative materials: a review. Wear. 2006 Nov 20;261(9):980-6.

123. Turssi CP, de Moraes Purquerio B, Serra MC. Wear of dental resin composites: insights into underlying processes and assessment methods—a review. Journal of Biomedical Materials Research Part B: Applied Biomaterials: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and The Australian Society for Biomaterials and the Korean Society for Biomaterials. 2003 May 15;65(2):280-5.

124. Dawes C, Pedersen AM, Villa A, Ekström J, Proctor GB, Vissink A, Aframian D, McGowan R, Aliko A, Narayana N, Sia YW. The functions of human saliva: A review sponsored by the World Workshop on Oral Medicine VI. Archives of oral biology. 2015 Jun 1;60(6):863-74.

125. ISO D. Guidance on testing of wear. Part 2: Wear by two-and/or three body contact.

126. Heintze SD, Reichl FX, Hickel R. Wear of dental materials: Clinical significance and laboratory wear simulation methods—A review. Dental materials journal. 2019 May 29;38(3):343-53.

127. Leinfelder KF. Wear patterns and rates of posterior composite resins. International dental journal. 1987 Sep;37(3):152.

128. Rossomando KJ, Wendt Jr SL. Thermocycling and dwell times in microleakage evaluation for bonded restorations. Dental Materials. 1995 Jan 1;11(1):47-51.

129. Tsujimoto A, Barkmeier WW, Fischer NG, Nojiri K, Nagura Y, Takamizawa T, Latta MA, Miazaki M. Wear of resin composites: Current insights into underlying mechanisms, evaluation methods and influential factors. Japanese Dental Science Review. 2018 May 1;54(2):76-87.

130. DeLong R, Pintado MR, Douglas WH, Fok AS, Wilder Jr AD, Swift Jr EJ, Bayne SC. Wear of a dental composite in an artificial oral environment: a clinical correlation. Journal of Biomedical Materials Research Part B: Applied Biomaterials. 2012 Nov;100(8):2297-306.

131. Heintze SD, Faouzi M, Rousson V, Özcan M. Correlation of wear in vivo and six laboratory wear methods. Dental Materials. 2012 Sep 1;28(9):961-73.; 961-73.

132. Barkmeier WW, Erickson RL, Latta MA, Lambrechts P. Comparison of laboratory and clinical wear rates of resin composites. Quintessence International. 2004 Apr 1;35(4).

133. Bayne SC. Correlation of clinical performance with 'in vitro tests' of restorative dental materials that use polymer-based matrices. Dental Materials. 2012 Jan 1;28(1):52-71.

134. Ruengrungsom C, Palamara JE, Burrow MF. Comparison of ART and conventional techniques on clinical performance of glass-ionomer cement restorations in load bearing areas of permanent and primary dentitions: A systematic review. Journal of dentistry. 2018 Nov 1;78:1-21.

135. Roulet JF, Hussein H, Abdulhameed NF, Shen C. In vitro wear of two bioactive composites and a glass ionomer cement. Dtsch Zahnärztl Z Int. 2019;1:24-30.

136. Latta MA, Tsujimoto A, Takamizawa T, Barkmeier WW. In Vitro Wear Resistance of Self-Adhesive Restorative Materials. J Adhes Dent. 2020 Jan 1;22:59-64.

137. Price RB, Ferracane JL, Shortall AC. Light-curing units: a review of what we need to know. Journal of Dental Research. 2015 Sep;94(9):1179-86.

138. Francois P, Fouquet V, Attal JP, Dursun E. Commercially Available Fluoride-Releasing Restorative Materials: A Review and a Proposal for Classification. Materials. 2020 Jan;13(10):2313.

139. Bansal R, Burgess J, Lawson NC. Wear of an enhanced resin-modified glass-ionomer restorative material. American journal of dentistry. 2016 Jun;29(3):171-4.

140. Moberg M, Brewster J, Nicholson J, Roberts H. Physical property investigation of contemporary glass ionomer and resin-modified glass ionomer restorative materials. Clinical Oral Investigations. 2019 Mar 8;23(3):1295-308.

141. Heintze SD, Reichl FX, Hickel R. Wear of dental materials: Clinical significance and laboratory wear simulation methods—A review. Dental materials journal. 2019 May 29;38(3):343-53.

142. Matias P, Roulet J-F, Abdulhameed N, Shen C: In vitro wear of 4 different universal composites. Stomatol Edu J 2016; 3: 70–77.

143. Palaniappan S, Bharadwaj D, Mattar DL, Peumans M, Van Meerbeek B, & Lambrechts P (2011) Nanofilled and microhybrid composite restorations: Five-year clinical wear performances *Dental Materials* **27(7)** 692-700.

144. Heintze SD, Barkmeier WW, Latta MA, Rousson V. Round robin test: wear of nine dental restorative materials in six different wear simulators–supplement to the round robin test of 2005. dental materials. 2011 Feb 1;27(2):e1-9.

145. Lohbauer U. Dental glass ionomer cements as permanent filling materials?–properties, limitations and future trends. Materials. 2010 Jan;3(1):76-96.